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# THE CRYSTAL STRUCTURES OF SOME ORGANO-METALLIC COMPOUNDS OF BERYLLIUM AND ZINC

A thesis submitted for the degree of
doctor of philosophy by G.W. Adamson, B.Sc.
of Hatfield College.

October, 1966.

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## SUMMARY

X-ray diffraction methods have been used to show the existence of $BeH_2Be$ bridges and to investigate the association, in the solid state, of alkylsulphides of methyl zinc.

The diethylether complex of sodium hydridodiethylberyllate forms monoclinic crystals with a = 5.044 Å, b = 11.17 Å, c = 20.90 Å, $\beta$ = 101°15' and space group $P2_1/c$. There are two units of $Na_2Et_4Be_2H_2 \cdot 2Et_2O$ in the unit cell. The crystal can be thought of as being made up of $[NaOEt_2]^+$ and $[Et_4Be_2H_2]^{2-}$ ions. Pairs of $[NaOEt_2]^+$ ions are related by the centre of symmetry at (0,0,0), the Na-Na distance being 3.620 Å and the Na-O distance 2.350 Å. The carbon atoms of the ether molecule appear to be disordered. The centrosymmetric $[Et_4Be_2H_2]^{2-}$ ions are positioned around the centre of symmetry at $(\frac{1}{2},0,0)$. The Be-Be distance is 2.221 Å. The two Be-C distances of 1.766 and 1.809 Å are significantly different from each other. The bridging hydrogen atom lies almost on the a axis, the mean Be-H and Na-H distances being 1.49 and 2.40 Å respectively. The Na-H-Na and Be-H-Be angles are the same within experimental error as are the H-Na-H and H-Be-H angles. There are some similarities in the environment of the bridging hydrogen atom with respect to the sodiums and berylliums.

Methylzinc tetiarybutylsulphide is monoclinic with a = 9.59 Å, b = 39.04 Å, c = 12.13 Å, $\beta$ = 116°8' and space group $P2_1/c$. There are 20 units of $MeZnSBu^t$ in the unit cell.

The compound exists in the crystal as descrete pentameric molecules, in agreement with the degree of association found from molecular weight measurements in benzene solution. The zinc atoms lie near the corners of a square-based pyramid with the apical atom, Zn5, closer to Zn3 and Zn4. S2, S3 and S4 lie above the centres of three of the triangular faces. S1 is above the face defined by Zn1, Zn2 and Zn5 but is much closer to Zn1 and Zn2. It is three-coordinate and pyramidal. S5 lies below the basal plane, is closer to Zn3 and Zn4 and is five-coordinate. All of the zinc atoms and S2, S3 and S4 are four-coordinate. There appears to be no appreciable Zn-Zn or S-S bonding. The Zn-S bonding distances range from 2.273 to 2.982 Å. The mean Zn-C , S-C and C-C bond lengths are 2.01, 1.87 and 1.57 Å respectively.

Methylzinc isopropylsulphide is tetragonal with $\underline{a}$ = 13.60 Å, $\underline{c}$ = 15.16 Å and space group $P4_2/nnm$. There are 16 units of $CH_3ZnSC_3H_7$ in the unit cell. In benzene solution the compound is hexameric but it must have a different degree of association in the solid. From the Patterson function it seems that atoms lie in special positions. Atomic positions which lead to a satisfactory agreement between observed and calculated structure factors have not yet been obtained.

Two computer programmes have been written. One correlates, by a least squares treatment of common reflections, intensity data which have been recorded about two or three crystal axes. The other programme will correct intensity data, which have been recorded by equi-inclination Weissenberg technique, for absorption, Lorentz and polarisation factors and spot length. The absorption correction can be computed for a crystal of any shape subject to the conditions that it must have plane surfaces and no re-entrant angles.

# ACKNOWLEDGEMENTS

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# INTRODUCTION

## Conditions for diffraction.

A crystal is an arrangement of atoms, ions or molecules which is periodic in three dimensions. If an arbitrary point is chosen as origin then that point and all other points with an identical environment form a space lattice. The lattice can be described in terms of three non-coplanar vectors, $\underline{a}$, $\underline{b}$, and $\underline{c}$. The parallelipiped formed by these vectors is named the unit cell. The crystal is generated by the action on the unit cell of operations involving translation alone.

Assume that there is one electron at each lattice point. Let A and B (Fig.1) be two lattice points related by the vector $\underline{r}$ such that

$$\underline{r} = x\underline{a} + y\underline{b} + z\underline{c} \qquad 1$$

where x, y and z are integers.

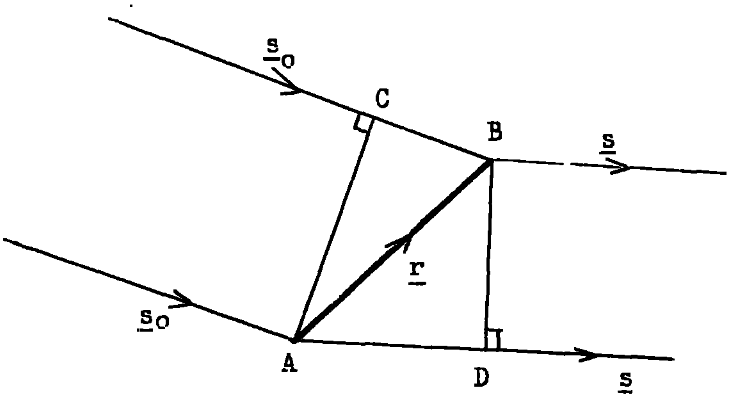


Fig.1.

If a beam of x-rays is incident on the lattice, each lattice point will act as a source of secondary radiation. Consider the resultant diffracted beam in the direction $\underline{s}$. Let the unit vectors in the direction of $\underline{s}_o$ be $\lambda\underline{s}_o$ and in the direction of $\underline{s}$ be $\lambda\underline{s}$ where $|\lambda\underline{s}_o| = |\lambda\underline{s}|$. The phase difference $\psi$ in the direction of $\underline{s}$ between the rays from A and B is

$$\psi = AD - CB$$

$$= \lambda(\underline{r} \cdot \underline{s} - \underline{r} \cdot \underline{s}_o)$$

where $\underline{r} \cdot \underline{s}$ is the scalar product of $\underline{r}$ and $\underline{s}$. Let $\underline{R} = \underline{s} - \underline{s}_o$.

$$\psi = \lambda\underline{r} \cdot \underline{R} \qquad 2.$$

Diffracted rays will have a non-zero intensity when

$$\psi = m\,(2\pi) \qquad 3.$$

$$= m\lambda$$

where m is integral and $\lambda$ is the wavelength of the incident X-rays. Eliminating $\psi$ from 2 and 3 gives $\underline{r} \cdot \underline{R} = m$

or $(x\underline{a} + y\underline{b} + z\underline{c}) \cdot R = m$

The above equation can be rewritten as three equations which are known as the 'Laue equations'.

$$\underline{a} \cdot \underline{R} = h$$

$$\underline{b} \cdot \underline{R} = k$$

$$\underline{c} \cdot \underline{R} = l$$

Where h, k and l are integers.

### The reciprocal lattice

The Laue equations can be rewritten to give three equations of the form $\dfrac{\underline{a}}{h} \cdot \underline{R} = 1$ 4.

If the equations 4 are taken in pairs then three equations of the type below are obtained.

$$\left(\frac{\underline{a}}{h} - \frac{\underline{b}}{k}\right) \cdot \underline{R} = 0 \qquad 5.$$

The three equations 5. describe the fact that (Fig.2.) the vector $\underline{R}$ is perpendicular to the plane PQR which has intercepts along the a,b and c axes of $\frac{\underline{a}}{h}$, $\frac{\underline{b}}{k}$ and $\frac{\underline{c}}{l}$ respectively. The vector $\underline{R}$ is thus in a direction normal to the plane of Miller indices hkl.

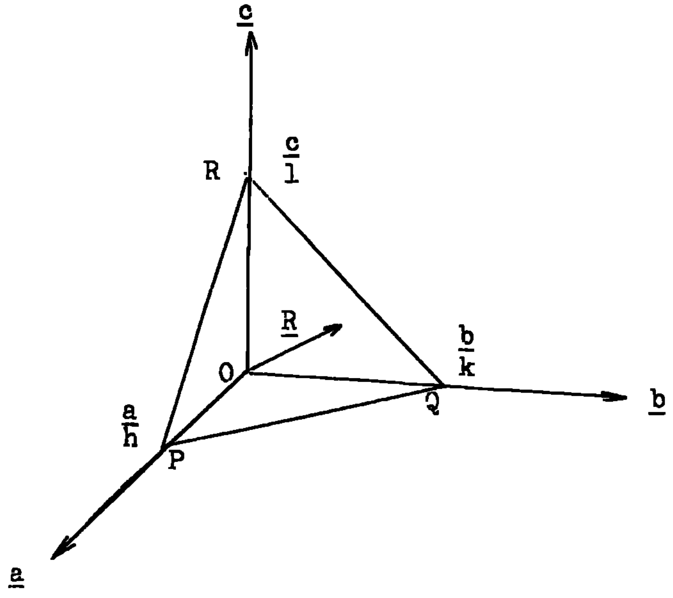

$\underline{c}$
R
$\frac{\underline{c}}{l}$
$\underline{R}$
O
$\frac{\underline{b}}{k}$
$\underline{b}$
Q
$\frac{\underline{a}}{h}$
P
$\underline{a}$

Fig.2.

If $\underline{s}_0$ makes an angle of $\theta$ with the plane (hkl), then as $|\underline{s}_0| = |\underline{s}|$, $\underline{R}$ must bisect the angle between the incident and diffracted rays. The diffracted ray can thus be regarded as having been reflected from the plane (hkl).

$\underline{R}$ may be described in terms of three non-coplanar vectors $\underline{a}^*$, $\underline{b}^*$ and $\underline{c}^*$

$$\underline{R} = \xi\underline{a}^* + \eta\underline{b}^* + \zeta\underline{c}^* \qquad 7.$$

If equation 7 is substituted into the Laue equations then the following equations result.

$$\underline{a}.\ (\xi\,\underline{a}^* + \eta\,\underline{b}^* + \zeta\,\underline{c}^*) = h$$
$$\underline{b}.\ (\xi\,\underline{a}^* + \eta\,\underline{b}^* + \zeta\,\underline{c}^*) = k$$
$$\underline{c}.\ (\xi\,\underline{a}^* + \eta\,\underline{b}^* + \zeta\,\underline{c}^*) = 1 \qquad 8.$$

The equations are only satisfied if

$\xi = h, \eta = k, \zeta = 1,$

$\underline{a}.\ \underline{a}^* = \underline{b}.\ \underline{b}^* = \underline{c}.\ \underline{c}^* = 1,$

and $\underline{a}.\ \underline{b}^* = \underline{a}.\ \underline{c}^* = \underline{b}.\ \underline{a}^* = \underline{b}.\ \underline{c}^* = \underline{c}.\ \underline{a}^* = \underline{c}.\ \underline{b}^* = 0$

The lattice defined by the vectors a*, b* and c* is known as the reciprocal lattice. Each point corresponds to a reflection from a plane with Miller indices (hkl), and is at a distance of $^1/d(hkl)$, along a vector in the direction of the normal to the plane (hkl).

## The structure factor

In the previous discussion the scattering was assumed to be by one electron at a lattice point. In crystals, atoms are positioned at various places in the unit cell, and the waves scattered by the different atoms will have different phases. The resultant intensity and phase of the scattered ray will be found by combining the waves from each atom.

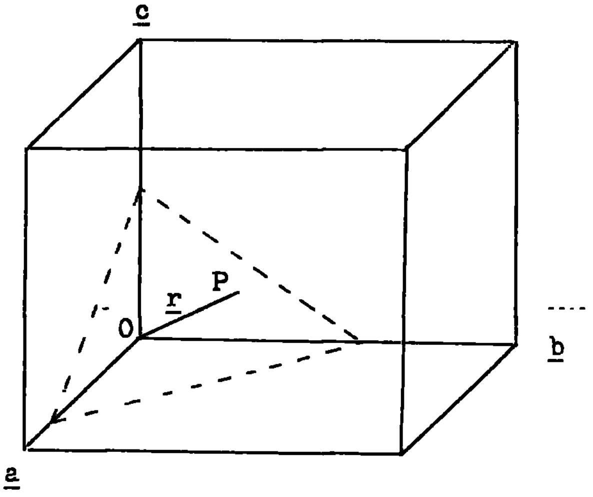


Fig.3.

In Fig.3 let the electron density be $\rho(xyz)$ at some point P in the unit cell where P is at the end of the vector.

$$\underline{r} = x\,\underline{a} + y\,\underline{b} + z\,\underline{c} \qquad 9$$

In equation 9; x, y and z are non-integral and are expressed as fractions of the unit-cell edge. The dotted line in Fig.3 represents a plane (hkl). The perpendicular from the origin to the plane corresponds to a phase change $2\pi$, this vector (see equation 7) is $\underline{R}$. The point P can be thought of as lying on a plane parallel to the plane (hkl). The projection of $\underline{r}$ on $\underline{R}$ will be the length of the normal from this plane which contains P to the origin.

The phase $\phi$ of the wave scattered by the volume element dV around P is given by

$$\frac{\phi}{2\pi} = \underline{R} \cdot \underline{r}$$

$$= (h\underline{a}^* + k\underline{b}^* + l\underline{c}^*) \cdot (x\underline{a} + y\underline{b} + z\underline{c})$$

$$\phi = 2\pi\ (hx + ky + lz).$$

The amplitude of this ray will be $\rho(xyz)\,dV$. The resultant scattered wave in the direction $\underline{s}$ is

$$F(hkl) = V \iiint_{0}^{1} \rho(xyz) \exp 2\pi i\ (hx + ky + lz).\ dxdydz.$$

F(hkl) is the structure factor corresponding to the plane of Miller indices (hkl).

The structure factor can be expressed in a way which is more convenient for computational purposes. The electronic distribution in atoms can be calculated and thus the atomic scattering factor $f_o$ can be found. This is the ratio of the scattering power of the atom compared with that of one electron. Because of interference between waves scattered by different parts of the same atom the scattering factor decreases with increasing $\theta$. The structure factor can be expressed as a summation, which is made over all N atoms in the unit cell.

$$F(hkl) = \sum_{j=1}^{N} f_j \exp 2\pi i\ (hx_j + ky_j + lz_j)$$

In the above equation $f_j$ is the scattering factors and $x_j$, $y_j$ and $z_j$ are the fractional coordinates of the $j^{th}$ atom. The quantity F(hkl) is complex and may be expressed in terms of its real and imaginary parts.

$$F(hkl) = \sum_{j=1}^{N} f_j \cos 2\pi\ (hx_j + ky_j + lz_j)$$

$$+\ i \sum_{j=1}^{N} f_j \sin 2\pi\ (hx_j + ky_j + lz_j)$$

or $F = A + i\,B$.

The modulus of the structure factor, $|F(hkl)| = \sqrt{A^2 + B^2}$ and the phase constant $\alpha(hkl) = \tan^{-1}(B/A)$.

The atoms in a crystal perform thermal vibrations at all temperatures. Corresponding atoms in different unit cells will not be at the same point in their oscillatory movement at the same time and so F(hkl) will be reduced by an amount which increases with $\theta$. This is allowed for by reducing the atomic scattering factor $f_o$ for an atom at rest by multiplying it by an exponential term.

$$\exp(-B\sin^2\theta/\lambda^2)$$

If $\bar{u}^2$ is the mean square displacement of an atom from a plane and the thermal motion is isotropic then

$$B = 8\pi^2\bar{u}^2$$

The thermal motion is in general not isotropic and is better described in terms of an anisotropic vibration. The vibrations are described by a symmetrical tensor $\underline{U}$ which has six independent components, such that the mean square amplitude of vibration in the direction of a unit vector $\underline{l} = (l_1, l_2, l_3)$ is $\bar{u}^2 = \sum_{i=1}^{3}\sum_{j=1}^{3} U_{ij}\, l_i\, l_j$.

The transform of the smearing function is

$$q(\underline{s}) = \exp\left[-2\pi^2\left(\sum\sum U_{ij}\, s_i\, s_j\right)\right]$$

where $\underline{s} = (s_1, s_2, s_3)$ is the reciprocal vector. If the units of $U_{ij}$ are $Å^2$ and those of $s_i$ are $Å^{-1}$ then at a reciprocal lattice point $\underline{s} = (ha^*, kb^*, lc^*)$.

$$q(hkl) = \exp\left[-2\pi^2\left(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{31}lhc^*a^* + 2U_{12}hka^*b^*\right)\right]$$

Fourier series (Duane, 1925; Havinghurst, 1925; Bragg, 1929)

The electron density of a crystal is periodic in three dimensions and it can be represented in terms of a Fourier series.

$$\rho(x,y,z) = \sum_{h',\,k',\,l' = -\infty}^{+\infty}\sum\sum C(h'k'l') \exp 2\pi i\,(h'x+k'y+l'z).$$

C is a Fourier coefficient and h', k' and l' are three indices which are allotted to it. If this expression for the electron density is substituted into the structure factor equation then the following results.

$$F(hkl) = \int\int_0^1\int \sum\sum_{-\infty}^{+\infty}\sum C(h'k'l') \exp 2\pi i\,(h'x+h'y+l'z)$$
$$.\exp 2\pi i\,(hx+ky+lx)\ V\ dx\ dy\ dz.$$

Both of the exponential functions are periodic and the integral of their product will be zero unless $h = -h'$, $k = -k'$ and $l = -l'$. In a crystal the integral cannot have a zero value so that the above condition must hold.

$$\text{Thus } F(hkl) = \int\int_0^1\int C(h'k'l')\ V\ dx\ dy\ dz,$$

$$\text{or}\quad C(h'k'l') = \frac{1}{V}\cdot F(hkl),$$

the Fourier coefficients are directly related to the structure factor, and the electron density function is

$$\rho\ (x,y,z) = \frac{1}{V}\sum_{hkl = -\infty}^{+\infty}\sum\sum F(hkl) \exp\left[-2\pi i\,(hx+ky+lz)\right]$$

$$\text{or } \rho(x,y,z) = \frac{1}{V}\sum_{hkl = -\infty}^{+\infty}\sum\sum \left|F(hkl)\right| \cos\left[2\pi(hx+ky+lz) - \alpha(hkl)\right]$$

where $\alpha(hkl)$ is a phase angle

The Patterson function (Patterson, 1935)

The electron density in a crystal at a point x,y,z is given by

$$\rho(x,y,z) = \frac{1}{V}\sum\sum_{h,k,l=-\infty}^{+\infty}\sum F(h\,k\,l)\exp -2\pi i\,(hx+ky+lz)$$

and at a point (x+u), (y+v), (z+w) by

$$\rho(x+u,\ y+v,\ z+w) = \frac{1}{V}\sum\sum_{h'k'l'=-\infty}^{+\infty}\sum F(h'k'l')\exp -2\pi i\left[h'(x+u)+k'(y+v)+l'(z+w)\right]$$

If the two electron density functions are multiplied together and integrated over the unit cell then the value of the Patterson function at a point (u,v,w,) is given by

$$P(u,\ v,\ w) = \int_0^1 \rho(x,y,z)\cdot\rho(x+u,\ y+v,\ z+w)\,dv.$$

The integrals of each of the products is zero except when $h = -h'$, $k = -k'$ and $l = -l'$.

Thus

$$P(u,v,w) = \frac{1}{V}\sum\sum_{h,k,l=-\infty}^{+\infty}\sum F(h\,k\,l).\,F(\bar{h}\,\bar{k}\,\bar{l})\exp 2\pi i\,(hu+kv+lw)$$

$F(h\,k\,l)$ and $F(\bar{h}\,\bar{k}\,\bar{l})$ are complex conjugates and hence their product equals $|F(h\,k\,l)|^2$. The Patterson function is calculated from the squares of the observed structure factors.

If there are atoms at (x,y,z) and (x+u, y+v, z+w) in the crystal then the Patterson function will have a maximum at (u,v,w).

The height of a peak will be equal to the product of the electron densities of the atoms causing it.

The heavy atom method (Robertson and Woodward, 1940)

The electron density distribution of a crystal cannot be computed directly from the observed structure factors as their phases are not experimentally observable quantities. An atom of high atomic number will give rise to large, easily identifiable peaks in the Patterson function from which its coordinates may be deduced. Structure factors may now be calculated based on these known parameters, and an electron density map computed using $|F_o|$ as Fourier coefficients, and giving them the phases of the Fc. From this electron density map improved parameters for the heavy atoms will be found and the positions of some or all of the lighter atoms in the crystal may also be found. This process can be continued until the positions of all of the atoms are known.

Structure refinement.

Two methods of refinement have been used in the work described in this thesis, namely the methods of successive Fourier syntheses and of least-squares (Hughes, 1941). Fourier methods are most useful when additional features of the structure have to be determined during refinement.

Structure factors are calculated and from these an electron density map is computed. The positions of the atoms are then determined from the electron density map. These improved positions are used to compute further structure factors. The atomic coordinates which are obtained from Fo syntheses are subject to errors due to termination of series effect as in practice only a finite number of terms are available. These can be obviated by calculating an Fc synthesis and applying a back shift correction.

If the coefficients used in a Fourier summation are of the form (Fo(hkl) - Fc(hkl) ) then the resulting electron density map shows the difference between the electron density in the crystal and that in the postulated structure (Cochran, 1951). From a difference synthesis corrections to positional and isotropic or anisotropic temperature factors can be found (Leung, Marsh and Schomaker, 1957). These parameters are not subject to series termination effects.

If the parameters defining a structure are $u_1, u_2, \ldots, u_n$, then the calculated structure factor can be rewritten as a function of these parameters,

$$F_c = f\,(u_1, u_2, \ldots, u_n)$$

and $$F_o = f\,(u_1 + \epsilon_1, u_2 + \epsilon_2, \ldots, u_n + \epsilon_n)$$

where $\epsilon_i$ are the shifts required to give the true structural parameters. The expression for $F_o$ can be expanded as a Taylor series. If the approximation is good then only the first term need be considered.

$$F_o = f\,(u_1, u_2, \ldots, u_n) + \sum_{i=1}^{n} \frac{\partial f(u_1, u_2, \ldots, u_n)}{\partial u_i}\,\epsilon_i$$

or $$(F_o + F_c) = \sum_{i=1}^{n} \frac{\partial F_c}{\partial u_i}\,\epsilon_i$$

Each Fo is subject to random errors. The theory of errors gives the result that the most acceptable set of $\epsilon_i$ is that which minimises the sum of the weighted squares of the discrepancies.

i.e. $$\sum_h w\left(|Fo| - |Fc|\right)^2 = \sum_h w\Delta^2$$

where the summation is carried out over all observed structure factors. The weight w allows for the reliability of the observation and its value should be chosen so that w(hkl) is equal to $1/\sigma^2(hkl)$, where $\sigma$ is the standard deviation in $|Fo(hkl)|$.

When the last two equations above are combined a set of equations known as the normal equations result.

$$\sum_h w\,\Delta\,\frac{\partial Fc}{\partial u_i} = \sum_h w\left(\frac{\partial Fc}{\partial u_i}\right)^2 \epsilon_i + \sum_h w\,\frac{\partial Fc}{\partial u_i}\left(\sum_{j\neq i}^{n}\frac{\partial Fc}{\partial u_j}\cdot\epsilon_j\right)$$

The computations involved in finding the $\epsilon_i$ can be simplified by making certain assumptions i.e. if the atoms are well resolved then some of the off-diagonal terms in the full matrix can be neglected (Cruickshank, Pilling, Bujosa, Lovell and Truter, 1961)

It has been shown (Cochran, 1948) that the methods for determining corrections to the atomic parameters by least squares method and by difference syntheses are equivalent if the least squares weights $w_h = 1/F_h$.

## Accuracy of parameters obtained from least squares refinement

The normal equations are of form

$$\sum_{i=1}^{n} \epsilon_i a_{ij} = b_j$$

in matrix notation. The variance of the parameter $u_i$ is given by ( Cruickshank and Robertson, 1953)

$\sigma^2( u_i ) = ( a^{-1} )_{ii}$, where $(a^{-1})_{ii}$ is a diagonal term of the matrix which is the inverse of the matrix whose elements are $a_{ij}$. In most cases however, only relative weights are known and the experimental standard deviation (e.s.d.) $S(u_i)$ is given by

$$S^2 (u_i) = (a^{-1})_{ii} \sum w \Delta^2 / m - n$$

where (m - n) is the number of degrees of freedom of the system i.e. the excess of the number of observation over the number of parameters. In the block diagonal approximation the variances of parameters may be estimated from the diagonal terms of the inverses of the block matrices, however, this may lead to an underestimation because of the neglect of some interactions (Hodgson and Rollet, 1963).

## COMPOUNDS CONTAINING HYDROGEN BRIDGES

In the compounds which are to be discussed in this chapter, hydrogen atoms are bonded to two metal atoms forming a bridge. In these compounds there are not sufficient valence electrons to provide two electrons for each bond if only localized electron pair bonds are formed. For this reason the compounds are sometimes referred to as being electron-deficient. The structures and bonding of some compounds of boron containing hydrogen bridges will be discussed so that comparisons may be made with compounds of beryllium.

### Boron Compounds

The structure of diborane has been investigated by several physical techniques. The rotation - vibration spectrum (Price, 1947), vibration spectrum (Longuet - Higgins and Bell. 1943), nuclear magnetic resonance spectrum (Schoolery, 1955), and x-ray diffraction (Smith and Lipscomb, 1965) and electron diffraction data (Hedberg and Schomaker, 1951) are all consistent with it having a hydrogen-bridged structure. The dimensions of the diborane molecule quoted below are from the electron diffraction study.

Each boron atom is bonded to two terminal hydrogen atoms, the terminal H - B - H angle being 120°. The two boron atoms and four terminal hydrogen atoms are coplanar. The two bridging hydrogen atoms lie between the boron atoms in a plane perpendicular to that of the terminal hydrogen atoms.

The terminal B - H bonds are 1.19 ± 0.03 Å in length and can be explained in terms of localised electron pair bonds. Nuclear magnetic resonance studies (Ogg, 1954) indicate that the bridging hydrogen atoms have larger negative charges than the terminal hydrogens.

There are four electrons available to bind the two bridging hydrogen atoms and the two boron atoms together. Several explanations of the bonding in the hydrogen bridge have been put forward. Longuet - Higgins (1949) proposed that the bonding should be explained in terms of three-centre molecular orbitals each covering both of the boron atoms and one of the bridging hydrogen atoms.

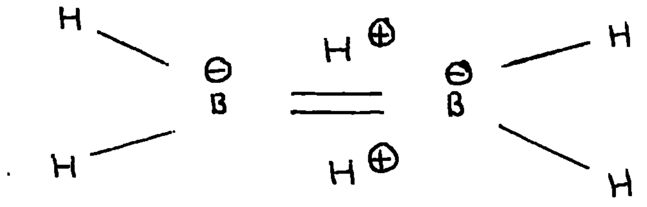
The 2s and 2p atomic orbitals of the boron atoms are assumed to be $sp^2$ hybridised. Two hybrid orbitals of each boron atom point towards a terminal hydrogen atom. Two hybrid orbitals are formed from the sum and difference of the $sp^2$ orbital which lies along the boron-boron direction and the $2p\pi$ boron atomic orbital, which lies in the plane of the two boron atoms and the two bridging hydrogen atoms. These hybrid orbitals point directly towards the bridging hydrogen atoms.

Two molecular orbitals can be formed, each from the 1s atomic orbital of one of the hydrogen atoms ($\psi_B$) and from one of the hybrid orbitals on each of the boron atoms, $\psi_A$ and $\psi_C$. Each molecular orbital, $\psi_M$, is of the form,

$$\psi_M = a(\psi_A + \psi_C) + \sqrt{1 - 2a^2}\,\psi_B$$

where 'a' is a normalising constant. Two electrons are placed in each molecular orbital. The bridging B - H distance is 1.33 ± 0.03 Å and the bridging H - B - H angle is 100°. Calculations using this model (Hamilton, 1956 and 1958; Yamazaki, 1957) show that the bridging hydrogen atoms carry negative charges of 0.22 electrons.

Pitzer (1945) describes the bridge in terms of a boron-boron double bond with two protons embedded in the resulting electron density.



Neither of these explanations is completely satisfactory (Coulson, 1961), the former because it does not suggest any direct boron-boron bonding. The boron-boron distance in diborane is 1.77 Å, which is 0.15 Å longer than that which would be expected for a boron-boron single bond on the basis of a covalent radius of 0.81 Å for boron. This corresponds to a bond order of 0.74 (Pauling, 1961). Molecular orbital calculations based on the three-centre model give a boron-boron bond order of 0.4 (Hamilton, 1956).

The higher boranes contain hydrogen bridges which may also be described in terms of multi-centre bonds. A detailed description of the boron hydrides is given by Lipscomb (1963).

The borohydrides of lithium and sodium are involatile solids, with ionic lattices made up of discrete cations and $BH_4^-$ anions. Proton resonance (Ford and Richards, 1955) and infrared spectroscopic (Price, 1949) studies show that in the $BH_4^-$ ion the hydrogen atoms have a tetrahedral distribution about the boron with a B - H distance of 1.25 ± 0.02 Å. This distance is greater by 1.7$\sigma$ than the terminal B - H distance in diborane. An increase in length would be expected because of the negative charge on the borohydride ion.

A partial X-ray structure analysis (Soldate, 1947) gives the Na - B distance in $NaBH_4$ as 3.07 Å. Price (1947) has calculated that the Na-H distance in $NaBH_4$ is 2.50 Å which may be compared with the Na-H distance of 2.440 ± 0.001 Å in sodium hydride. (Brewer and Mastick, 1951). Harris and Meihbohm (1947) obtained Li-B distance of 2.56 Å and 2.47 Å from a partial X-ray structure analysis of $LiBH_4$.

In contrast to $NaBH_4$ and $LiBH_4$, beryllium and aluminium ....

borohydrides are volatile, insoluble in polar solvents and violently hydrolysed by cold water at room temperature. Price (1949) combined the known intermetallic distances in the borohydrides with infrared measurements and calculated the metal-hydrogen distances. In $Be(BH_4)_4$ and $Al(BH_4)_3$ hydrogen atoms lie in bridging positions between the two different metal atoms. The distance from the boron atom to the bridging hydrogen atom is greater than the terminal B - H distance. It has been suggested (Longuet - Higgins, 1946) that there is a continuity in metal-hydrogen bond type from the more covalent interaction in diborane, through the mixed covalent-ionic interaction in $Be(BH_4)_2$ and $Al(BH_4)_3$ to the ionic structures of $LiBH_4$ and $NaBH_4$

## Beryllium Compounds

Several compounds have been prepared which contain beryllium-hydrogen bonds, but little structural information is available.

Beryllium hydride, in contrast to diborane which is a gas at room temperature, is an involatile insoluble solid. Several different methods of preparation have been reported (Barbaras, Dillard, Finholt, Wartik, Wilzbach and Schlesinger, 1951; Coates and Glockling, 1954, Banford and Coates, 1966) but none has yet yielded a crystalline sample. The compound is thought to be polymeric with bridging hydrogen atoms and its physical properties suggest that the bonding in it is more ionic than in diborane. This would be expected as beryllium is more electropositive than boron.

Beryllium atoms bridged by hydrogen atoms are known to be present in some compounds of formula $M_2Et_4Be_2H_2$ (Coates and Cox, 1962; Wittig and Hornburger, 1952) where M is an alkali metal and R is an organic group. Some of these compounds crystallise as ether complexes. While the X-ray structure analysis of $Na_2Et_4Be_2H_2.2Et_2O$ was in progress, Bell and Coates (1965) assigned bands in the infrared spectrum of the unsolvated compound to the $BeH_2Be$ group. If these compounds contain discrete anions of formula $(R_4Be_2H_2)^{2-}$ then these anions are isoelectronic with tetra-alkyl diboranes.

Ethylberyllium hydride has been prepared (Bell and Coates, 1966,a) but not isolated in pure form. Bell and Coates have found that certain compounds containing beryllium-hydrogen bonds will reduce unsaturated organic groups giving rise to reactions comparable to the hydroboration reactions.

The compound $Na_2BeH_4$ has been prepared (Bell and Coates, 1966,b), which is analagous to $NaBH_4$. It is reactive to oxygen and water and is insoluble in solvents with which it does not react. It is thought to be an electron-deficient polymer containing highly polarised metal-hydrogen bonds.

# THE CRYSTAL STRUCTURE OF THE DIETHYLETHER COMPLEX OF SODIUM HYDRIDODIETHYLBERYLLATE

## Introduction

The work to be described was carried out in order to obtain direct evidence for the existence of $\mathrm{BeH_2Be}$ bridges and to ascertain details of their structure. While this structure analysis was in progress Bell and Coates (1965) investigated the infrared spectra of some compounds thought to contain $\mathrm{BeH_2Be}$ bridges and assigned bands to absorptions due to this group.

Sodium hydridodiethylberyllate is a solid with a melting point of 198°C (Coates and Cox, 1962). It is soluble in diethylether from which it crystallises as colourless needle shaped crystals. The compound is stable at room temperature in the absence of oxygen and water, The presence of sodium atoms in the crystal made it possible to apply heavy atom methods with a reasonable chance that they would be successful. Initially it was thought that the substance whose structure was being examined was of formula $\mathrm{Na_2Et_4Be_2H_2}$. However during the course of the analysis it became apparent that the compound was the ether complex $\mathrm{Na_2Et_4Be_2H_2.2Et_2O}$

## Experimental

### Preparation of Crystals and Preliminary X-ray Examination

The crystals inflame immediately in air and react violently with water. They were prepared by heating sodium hydride with diethylberyllium in diethylether solution under reflux, in an atmosphere of dry nitrogen. The resulting solid was transferred to a Soxhlet apparatus and extracted with diethylether when crystals formed in the receiver. The crystals were picked out of the mother liquor in the dry nitrogen atmosphere of a glove-box and immediately sealed in pyrex glass capillary tubes. The crystals were needles, elongated along the a axis.

Some crystals were filtered from the solution, washed with diethylether, pumped under reduced pressure to remove excess solvent and then stored in a sealed glass tube. A density determination was later carried out on these crystal by floatation in a mixture of benzene and benzotrifluoride.

The unit cell dimensions were determined from precession photographs of the h0l, h1l, and hk0 reciprocal lattice nets taken with Mo $K\alpha$ radiation. The standard deviations of the unit cell lengths are of a 0.006 Å, of b 0.02 Å and of c 0.03 Å. However if systematic errors are included, the uncertainty is probably of the order of 0.25% The standard deviation on $\beta$ is about 10'.

The crystals were monoclinic.

$a = 5.044$, $b = 11.17$, $c = 20.90$ Å, $\beta = 101^\circ\ 15'$.

The conditions limiting observed reflections were

h0l, $l = 2n$.

0k0, $k = 2n$.

Space group $P2_1/c$,
$d_m = 1.04$ g. cm$^{-3}$

With four units of $Na_2Et_4Be_2H_2$ per unit cell
$d_x$ would be 1.052 g. cm$^{-3}$.

## Data Collection and Correction

The crystal used for data collection was a tapering needle of mean cross-section 0.35 x 0.44 mm$^2$. The reciprocal lattice nets 0kl, 1kl, 2kl, 3kl and 4kl were recorded photographically using multifilm equi-inclination Weissenberg technique and Cu. Kα radiation. The reciprocal lattice nets h0l, h1l, h2l and h3l were recorded photographically using timed exposures on a precession camera and Mo Kα radiation.

The intensities were estimated visually by comparison with a graduated scale and were corrected for Lorentz and polarization factors. The lengths of reflections on the upper net Weissenberg photographs were found to vary with their positions across the film as well as with $\zeta$ and $\xi$. The variation of spot-length with $\zeta$ and $\xi$ was allowed for by using the expression derived by Phillips (1956). The variation of spot-length across the films was allowed for by measuring the spot-lengths of a number of reflections of similar intensity and with similar $\zeta$ and $\xi$ coordinates. The lengths of other reflections were found by interpolation. No correction was made for absorption.

The data were put on the same relative scale by a least squares treatment of the common reflections.

## Structure Determination

The intensities were multiplied by a weighting function w where

$$\omega = \exp\left(\frac{12\,\mathrm{Sin}^2\theta}{\lambda^2}\right)$$

The function was not allowed to take a value greater than 100 so that too much importance was not given to reflections with large sin$\theta$ values. The magnitudes of these intensities are small, and thus subject to large errors in measurement.

The Patterson function was computed using as coefficients the weighted intensities prepared above. Each asymmetric unit of the unit cell was thought to contain one unit of $Na_2Et_4Be_2H_2$ thus the positions of two sodium ions were to be found. The major features of the Patterson function were easily interpreted on the basis of there being one sodium ion, NaI, in the asymmetric unit. Another sodium ion, Na2, could only be placed in the asymmetric unit by using smaller peaks in the Patterson function.

Further atomic coordinates were obtained by an application of the superposition method. The origin of the Patterson function was placed at the position of the single weight peak due to the vector between NaI and the atom related to it by the centre of symmetry at the origin. The superposition was carried out over two assymmetric units.

Some of the resulting regions of overlap were eliminated by a comparison of the two asymmetric units. This gave six large regions of overlap and several smaller ones.

Two of the large regions of overlap were at the positions chosen for the two sodium ions and were near the centre of symmetry at (0,0,0). Five of the other regions of overlap were near the centre of symmetry at ($\frac{1}{2}$,0,0) and together with the five positions related by this centre of symmetry were identified as being due to the carbon and beryllium atoms of an ($Et_4Be_2H_2$) unit. The beryllium atom was placed at a smaller region of overlap, and the four carbon atoms at the remaining four larger regions. Positions in the asymmetric unit had been given to two sodium atoms and to the atoms of one half of an ($Et_4Be_2H_2$) unit and so one half of an ($Et_4Be_2H_2$) unit was still to be located. The space near the centres of symmetry was occupied and thus the remaining atoms had to be in the region of the $2_1$ screw axes. The only other regions of overlap were near Na2 and they could not be understood in terms of ($Et_4Be_2H_2$) units.

Structure factors were calculated based on the two sodium ions, four carbon atoms, henceforth C1, C2, C3 and C4, and the beryllium atom. The residual was 0.475. An electron density map was computed and showed peaks at the positions to which atoms had been assigned.

The height of the peak due to Na2 was about 20% lower than that of the peak due to NaI, and near Na2 were four broad peaks of height about $2.0e.\text{Å}^{-3}$. No other peaks of height greater than $0.4e.\text{Å}^{-3}$ were observed.

It was not found possible to reconcile the electron density map and Patterson function with the chemical formula and unit cell contents given above. It was deduced that the compound was an ether complex of formula $Na_2Et_4Be_2H_2 . 2Et_2O$, with two formula units per unit cell. The asymmetric unit thus contained one less sodium atom, one less beryllium atom and one less bridging hydrogen atom, than had previously been thought. It did contain an oxygen atom which is located at the position given to Na2.

The chemical constitution of the compounds was reinvestigated (Bell and Coates, 1965). It was found to be an ether complex with a dissociation pressure of 17 mm. Hg. at 20°C. This ether is lost during the normal process of removing excess solvent by pumping at reduced pressure after crystallisation, and its presence had not been detected previously. In the case of the crystals which were used in this structure analysis, the precautions taken to avoid attack by oxygen and moisture also prevented the loss of ether. Crystals which have lost ether retain their shape, but do not give a sharp X-ray diffraction pattern.

The density of some freshly prepared crystals, which had not been subjected to reduced pressure was determined.

Dm = 0.958, Dx = 0.955 g. cm$^{-3}$

Z = 2 units of $Na_2Et_4Be_2H_2.2Et_2O$

F(000) = 368 electrons.

Linear absorption coefficient, Cu. K$\alpha$. radiation = 8.49 cm$^{-1}$.

## Structure Refinement

The structure was refined by five successive Fourier syntheses with adjustment of positional and isotropic temperature parameters. This reduced the residual to 0.296. Six cycles of least squares refinement were then computed with isotropic temperature factors. This produced a residual of 0.214. Four cycles of refinement with anisotropic temperature factors for each atom were calculated giving a residual of 0.186.

At this stage, an electron density difference map showed peaks of height 0.3 to 0.6e.Å$^{-3}$ due to the hydrogen atoms which are bonded to C1, C2, C3 and C4. Positions for these hydrogen atoms were calculated assuming tetrahedral H-C-H angles a C-H bond length of 1.09Å and a staggered arrangement for the methyl hydrogen atoms with respect to the methylene hydrogens. The observed hydrogen positions were very close to the calculated ones. The positions quoted in this thesis are those which were obtained from the difference map.

In the region where the bridging hydrogen atom was expected, a well shaped peak of height 0.5eÅ$^{-3}$ was observed. This hydrogen atom was designated H11.

Up to this point the carbon atoms of the ether molecule had refined with large temperature factors and the molecular dimensions had differed considerably from those expected. Features on the difference map could be interpreted as arising from disorder in the positions of the carbon atoms of the ether molecule.

The hydrogen atoms, H1 to H 10, bonded to the methyl and methylene carbon atoms of the ethyl groups attached to beryllium were given isotropic temperature factors of U = 0.15 and 0.11Å$^2$ respectively. These values were chosen because they were 50% greater than the values of the temperature parameters of the carbon atoms to which they were bonded, as obtained from the last cycle of isotropic refinement. The inclusion of contributions due to the hydrogen atoms H1 to H11 in the structure factors lowered the residual to 0.16.

The hydrogen atoms of the ether molecule were not located. The contributions of the alkyl hydrogen atoms H1 to H10 were included in structure factor calculations from this point but their parameters were not refined. The other atoms including H11, the bridging hydrogen atom, were given anisotropic temperature factors in the subsequent refinement.

Both the ordered and disordered arrangements were refined further so that a choice could be made between them. The parameters used in the refinement of the disordered model were identical with those used in the case of the ordered model except that the four carbon atoms of the ether molecule, C5, C6, C7 and C8 were replaced by eight carbon atoms, C5a, C5b, C6a, C6b, C7a, C7b, C8a, and C8b which were given occupation numbers of 0.5.

Five cycles of refinement were carried out on both ordered and disordered arrangements. For the disordered model residuals of $R = \dfrac{\sum w\left||Fo| - |Fc|\right|}{\sum w|Fo|}$

$= 0.133$ and $R' = \dfrac{\sum w\left||Fo| - |Fc|\right|^2}{\sum w|Fo|^2} = 0.041$

were obtained. In the case of the ordered model the residuals R and R' were reduced to 0.143 and 0.045. In both cases, on the fifth cycle of refinement the parameter shifts were less than their corresponding e.s.d.'s.

The significance of this difference in residuals was assessed using the tests given by Hamilton (1965). In the refinement, 1101 independent structure factors were used and the difference in the numbers of parameters which were refined in the two cases was 36. For the disordered model 146 parameters were refined and thus the number of degrees of freedom was 955. There is a 99.5% probability that the disordered model is a better representation of the actual arrangement than the ordered one.

The structure with ordered ether carbon atoms was rejected in favour of the disordered model.

Up to this point refinement had been carried out by the block diagonal approximation, using a 9 x 9 matrix to calculate shifts in the positional and anisotropic thermal parameters of each atom. In the disordered structure, the carbon atoms in the ether molecule had large temperature factors, and some of them were within 1Å of each other. It was thought that full matrix refinement might produce more accurate parameters for these atoms. From this point in the structure analysis, the ether carbon atoms were refined using a full matrix least squares programme, each atom having anisotropic temperature factors. Two cycles of least squares refinement were carried out, in each case the parameters refined were those of four atoms representing one disordered ethyl group. These produced no large parameter shifts. The e.s.d's of the disordered atoms however were larger than those previously obtained using the block diagonal approximation.

The indexing of some high order reflections was checked. To allow for the effects of the changes in the parameters of the disordered atoms, two cycles of least squares refinement using the block diagonal approximation were computed without refinement of these parameters. The parameters of each of the disordered ethyl groups were then given two cycles of full matrix refinement.

Finally the parameters of Na, Be, O, C1, C2, C3, C4, and H11 were refined through two cycles of least squares calculations, using the block diagonal approximation.

Final Cycle of Refinement

$$\sum |Fo| = -8108, \quad \sum |Fc| = 7735, \quad \sum \Delta = 951, \quad R = 0.117.$$

$$\sum w|Fo|^2 = 214, \quad \sum w|Fc|^2 = 207, \quad \sum w\Delta^2 = 5.5, \quad R' = 0.026.$$

Refinement was carried out over 1094 independent structure factors. Unobserved reflections were given zero weight in the refinement.

The structure factors were weighted by the function $\sqrt{w}$ where $\sqrt{w} = 1/(32 + |Fo| + 2.4\,|Fo|^2)^{\frac{1}{2}}$.

The atomic scattering factors used were those given in "InternationalTables for X-ray Crystallography", Volume III.

The scattering curve used for sodium was that of $Na^+$ and for the bridging hydrogen atom that of $H^-$.

On the last cycle of refinement, no parameter shift was greater than 0.57 of its e.s.d.

The final values of the atomic coordinates and their e.s.d's. are given in Table I. The final values of the thermal parameters and their e.s.d's. are given in Table 2. The correlation coefficients, $a_{12}$, $a_{13}$ and $a_{23}$, are given in Table 3.

## Final Difference Map

An difference electron density map was computed using the final values of the structure factors. This showed no pronounced features near any of the atomic positions.

## Description of the Structure

The arrangement of the atoms along the direction of the a axis, is shown in Fig. 1. Sodium ions are arranged in pairs about the centre of symmetry at (0,0,0) and the beryllium atoms in pairs about the centre of symmetry at ($\frac{1}{2}$,0,0). The pairs of beryllium atoms are bridged by hydrogen atoms which lie almost on the a axis. The bridging hydrogen atoms are surrounded by two beryllium atoms and two sodium ions which lie at the corners of a distorted tetrahedron. An ether molecule is coordinated to each sodium ion by its oxygen atom, and two ethyl groups are bonded to each beryllium atom. The bonding interactions are confined to chains which run through the crystal in a direction parallel to the a axis. The structure can be most simply described as being built up of $(NaOEt_2)^+$ and $(Et_4Be_2H_2)^{2-}$ ions.

The bond lengths and angles are shown in Table 4 and Table 5 respectively. The e.s.d.'s of the bond lengths and angles were calculated using the correlation coefficients $a_{13}$, which are shown in Table 3. No allowance was made for the effect of errors in the unit cell dimensions. In Fig.1. the carbon atoms of the ether molecules are omitted, and in the region of the bridging hydrogen atoms the drawing is distorted slightly. In the tables, figures and following discussion the coordinates of an atom whose symbol is primed (e.g. Na') is related to the atom whose coordinates are given in Table 1 by the operration of the centre of symmetry at (0,0,0).

Double priming of a symbol denotes that the coordinates are operated on by the centre of symmetry at $(\frac{1}{2},0,0)$, and Na''' is related to Na by a translation of one unit cell length along a. The environments of the sodium, beryllium and bridging hydrogen atoms are shown in Fig.2. The distance Na-Na' is 3.620 ± 0.004 Å, which is shorter than the distance found in sodium metal, but longer than the distance of 3.078Å in the $Na_2$ molecule in the gas phase (Herzberg, 1950). The Be-Be'' distance of 2.219 ± 0.013 Å is the same as that found in metallic beryllium (2.226 Å). The four Na-Be distances range from 3.051 ± 0.006 Å to 3.674 ± 0.006 Å, which can be compared with the value of 2.97 Å for the sum of their respective metallic radii. In diborane the B-B distance is 0.15 Å longer than that expected for a B-B single bond and corresponds to a bond order of 0.6 (Pauling, 1960). In $Me_6Al_2$ (Lewis and Rundle, 1953) the Al-Al distance of 2.55 Å is 0.03 Å longer than that expected for an Al-Al single bond. The Be-Be distance in $(Me_2Be)n$ (Rundle and Snow, 1951) is 2.11 Å and corresponds to a bond order of 0.31. Thus in the bridged compounds mentioned above, the direct Be-Be interaction is weaker than that between boron or aluminium atoms.

## $Na_2Et_4Be_2H_2.2Et_2O.$ TABLE 1

### Final values of Atomic Coordinates and their Standard Deviations

| Atom | $^x/a$ | $^y/b$ | $^z/c$ | $\sigma(^x/a)$ | $\sigma(^y/b)$ | $\sigma(^z/c)$ |
|---|---|---|---|---|---|---|
| Na | 0·08513 | 0·11904 | 0·05921 | 0·00042 | 0·00020 | 0·00011 |
| O | 0·11016 | 0·25845 | 0·14438 | 0·00087 | 0·00037 | 0·00019 |
| C1 | 0·7239 | 0·1257 | -0·1469 | 0·0013 | 0·0006 | 0·0003 |
| C2 | 0·5181 | 0·0509 | -0·1183 | 0·0010 | 0·0005 | 0·0003 |
| C3 | 0·5474 | 0·2252 | -0·0039 | 0·0010 | 0·0004 | 0·0003 |
| C4 | 0·3445 | 0·3137 | -0·0422 | 0·0012 | 0·0006 | 0·0003 |
| C5a | 0·0463 | 0·1569 | 0·2353 | 0·0064 | 0·0038 | 0·0024 |
| C5b | 0·1921 | 0·1567 | 0·2460 | 0·0073 | 0·0044 | 0·0026 |
| C6a | 0·1041 | 0·2609 | 0·2109 | 0·0088 | 0·0040 | 0·0017 |
| C6b | -0·0283 | 0·2187 | 0·1914 | 0·0058 | 0·0057 | 0·0015 |
| C7a | 0·0370 | 0·3857 | 0·1230 | 0·0046 | 0·0026 | 0·0022 |
| C7b | 0·2213 | 0·3688 | 0·1510 | 0·0111 | 0·0025 | 0·0017 |
| C8a | 0·2963 | 0·4618 | 0·1383 | 0·0048 | 0·0028 | 0·0016 |
| C8b | 0·1382 | 0·4446 | 0·1128 | 0·0108 | 0·0035 | 0·0015 |
| Be | 0·5171 | 0·0734 | -0·0347 | 0·0010 | 0·0006 | 0·0003 |
| H1 | 0·699 | 0·120 | -0·196 |  |  |  |
| H2 | 0·924 | 0·088 | -0·133 |  |  |  |
| H3 | 0·700 | 0·212 | -0·133 |  |  |  |

.... Cont.

**TABLE 1** (cont.)

| | | | | | | |
|---|---|---|---|---|---|---|
| H4 | 0·337 | 0·076 | -0·150 | | | |
| H5 | 0·512 | -0·037 | -0·134 | | | |
| H6 | 0·725 | 0·262 | -0·007 | | | |
| H7 | 0·549 | 0·237 | -0·045 | | | |
| H8 | 0·149 | 0·276 | -0·041 | | | |
| H9 | 0·375 | 0·400 | -0·032 | | | |
| H10 | 0·374 | 0·301 | -0·091 | | | |
| H11 | 0·3002 | 0·0000 | -0·0104 | 0·0063 | 0·0025 | 0·0017 |

## $Na_2Et_4Be_2H_2 \cdot 2Et_2O$. TABLE 2

### Final Values of Anisotropic Temperature Parameters ($\text{Å}^2$) and their Standard Deviations
### ($\text{Å}^2 \times 10^4$)

| **Atom** | $U_{11}$ | $U_{22}$ | $U_{33}$ | $2U_{23}$ | $2U_{31}$ | $2U_{12}$ |
|---|---|---|---|---|---|---|
| Na | 0·0794(14) | 0·0938(15) | 0·1056(15) | −0·0570(23) | 0·0139(23) | 0·0246(20) |
| O | 0·1148(30) | 0·1065(28) | 0·0949(27) | −0·0593(43) | 0·0726(45) | −0·0066(44) |
| C1 | 0·0942(45) | 0·1310(52) | 0·0996(43) | −0·0216(72) | 0·0997(70) | 0·0058(68) |
| C2 | 0·0662(29) | 0·0951(35) | 0·0817(32) | 0·0067(52) | 0·0452(49) | −0·0093(50) |
| C3 | 0·0642(29) | 0·0734(28) | 0·0981(36) | −0·0027(51) | 0·0485(50) | 0·0097(45) |
| C4 | 0·0871(39) | 0·0892(40) | 0·1322(52) | 0·0015(68) | 0·0521(71) | −0·0126(62) |
| C5a | 0·1435(225) | 0·1354(161) | 0·1561(322) | 0·0526(324) | 0·1041(506) | −0·0157(392) |
| C5b | 0·1963(347) | 0·1387(196) | 0·1719(247) | 0·0132(316) | 0·0385(632) | −0·0376(561) |
| C6a | 0·2377(425) | 0·1799(260) | 0·1131(185) | −0·0420(331) | 0·0546(438) | −0·0342(566) |
| C6b | 0·1260(186) | 0·3120(506) | 0·1198(215) | 0·0445(540) | 0·0736(298) | 0·0410(475) |
| C7a | 0·0760(118) | 0·1080(150) | 0·2362(315) | −0·0991(314) | 0·0518(292) | −0·0308(208) |
| C7b | 0·2256(359) | 0·1170(195) | 0·2569(314) | −0·0979(383) | −0·1878(508) | −0·0056(440) |
| C8a | 0·0959(140) | 0·1349(202) | 0·1981(249) | −0·0043(300) | 0·0592(275) | −0·0770(243) |
| C8b | 0·1608(347) | 0·1420(231) | 0·1552(195) | −0·0042(350) | 0·0288(422) | 0·0157(514) |
| Be | 0·0379(28) | 0·0757(37) | 0·0874(43) | −0·0112(60) | 0·0470(53) | −0·0079(52) |

.....Cont.

TABLE 2 (Cont.)

| | | | | | | |
|---|---|---|---|---|---|---|
| H11 | 0·0199(254) | -0·0074(160) | 0·0503(254) | 0·0016(335) | 0·0472(406) | 0·0685(325) |

H1, H2, H3, H8, H9 and H10 were given isotropic thermal parameters of 0·15Å$^2$.

H4, H5, H6 and H7 were given isotropic thermal parameters of 0·17Å$^2$.

## $Na_2Et_4Be_2H_2.2Et_2O$. TABLE 3

### Correlation Coefficients

| **Atom** | $a_{12}$ | $a_{13}$ | $a_{23}$ |
|---|---|---|---|
| Na | 0·10505 | 0·06460 | -0·22079 |
| O | -0·00949 | 0·25556 | -0·20287 |
| C1 | 0·00079 | 0·34705 | -0·05413 |
| C2 | -0·03456 | 0·22445 | 0·01180 |
| C3 | 0·03760 | 0·21496 | -0·00892 |
| C4 | -0·03637 | 0·17433 | -0·00959 |
| C5a | -0·01313 | 0·29189 | 0·14581 |
| C5b | 0·08788 | 0·06066 | 0·06174 |
| C6a | -0·31028 | 0·07087 | -0·47632 |
| C6b | 0·06863 | -0·03915 | -0·18933 |
| C7a | -0·44191 | 0·40722 | -0·61805 |
| C7b | 0·42658 | -0·57951 | -0·50511 |
| C8a | -0·52241 | 0·26586 | -0·27932 |
| C8b | 0·45014 | 0·30850 | 0·03884 |
| Be | -0·04142 | 0·26813 | -0·05099 |
| H11 | 0·31440 | 0·24454 | 0·02640 |

## $Na_2Et_4Be_2H_2 \cdot 2Et_2O$. TABLE 4.

### Bond lengths and their Standard Deviations

| | | | | | |
|---|---|---|---|---|---|
| Na-Na' | 3·620Å | ± 0·004Å | Be-H11 | 1·53 | 0·03 |
| Na-O | 2·350 | 0·005 | Be'-H11 | 1·44 | 0·03 |
| Na-H11 | 2·38 | 0·03 | Be-Be" | 2·219 | 0·013 |
| Na'-H11 | 2·41 | 0·03 | C1-H1 | 1·02 | |
| O-C6a | 1·397 | 0·037 | C1-H2 | 1·08 | |
| O-C6b | 1·385 | 0·036 | C1-H3 | 1·02 | |
| O-C7a | 1·514 | 0·031 | C2-H4 | 1·07 | |
| O-C7b | 1·350 | 0·034 | C2-H5 | 1·03 | |
| C1-C2 | 1·542 | 0·008 | C3-H6 | 1·00 | |
| C3-C4 | 1·532 | 0·008 | C3-H7 | 1·03 | |
| C2-Be | 1·766 | 0·008 | C4-H8 | 1·08 | |
| C3-Be | 1·810 | 0·008 | C4-H9 | 0·99 | |
| C5a-C6a | 1·32 | 0·06 | C4-H10 | 1·07 | |
| C5b-C6b | 1·59 | 0·06 | | | |
| C7a-C7b | 1·54 | 0·04 | | | |
| C8a-C8b | 1·18 | 0·05 | | | |

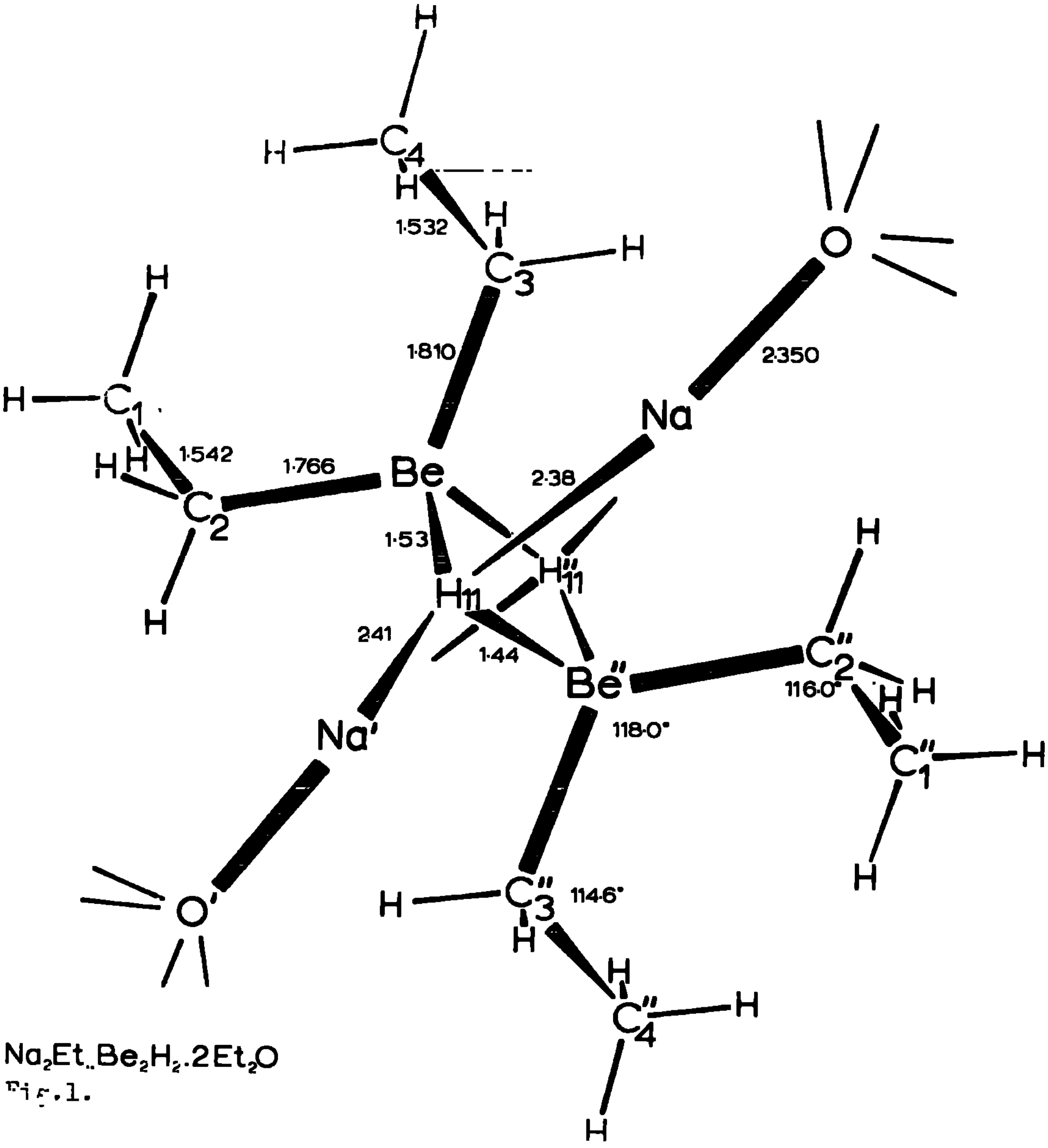
## $Na_2Et_4Be_2H_2 \cdot 2Et_2O$. TABLE 5.

## Bond Angles and their Standard Deviations

| Atoms | Angle | e.s.d. |
|---|---|---|
| Na'-Na-O | 167·2° | 0·2° |
| O-Na-H11 | 150·3 | 0·8 |
| O-Na-H11' | 127·3 | 0·8 |
| H11-Na-H11' | 81·9 | 1·1 |
| Na-O-C6a | 139·4 | 1·9 |
| Na-O-C6b | 111·4 | 2·3 |
| Na-O-C7a | 115·1 | 1·7 |
| Na-O-C7b | 130·0 | 1·9 |
| C6a-O-C7a | 112·6 | 2·8 |
| C6b-O-C7b | 118·5 | 3·0 |
| C2-C1-H1 | 114 | |
| C2-C1-H2 | 110 | |
| C2-C1-H3 | 106 | |
| H1-C1-H2 | 100 | |
| H1-C1-H3 | 110 | |
| H2-C1-H3 | 117 | |
| C1-C2-Be | 116·0 | 0·4 |
| C1-C2-H4 | 100 | |
| C1-C2-H5 | 112 | |
| Be-C2-H4 | 114 | |
| Be-C2-H5 | 116 | |
| H4-C2-H5 | 95 | |
| C4-C3-Be | 114·6 | 0·4 |
| C4-C3-H6 | 103 | |
| C4-C3-H7 | 108 | |
| Be-C3-H6 | 112 | |
| Be-C3-H7 | 117 | |
| H6-C3-H7 | 100 | |
| C3-C4-H8 | 105 | |

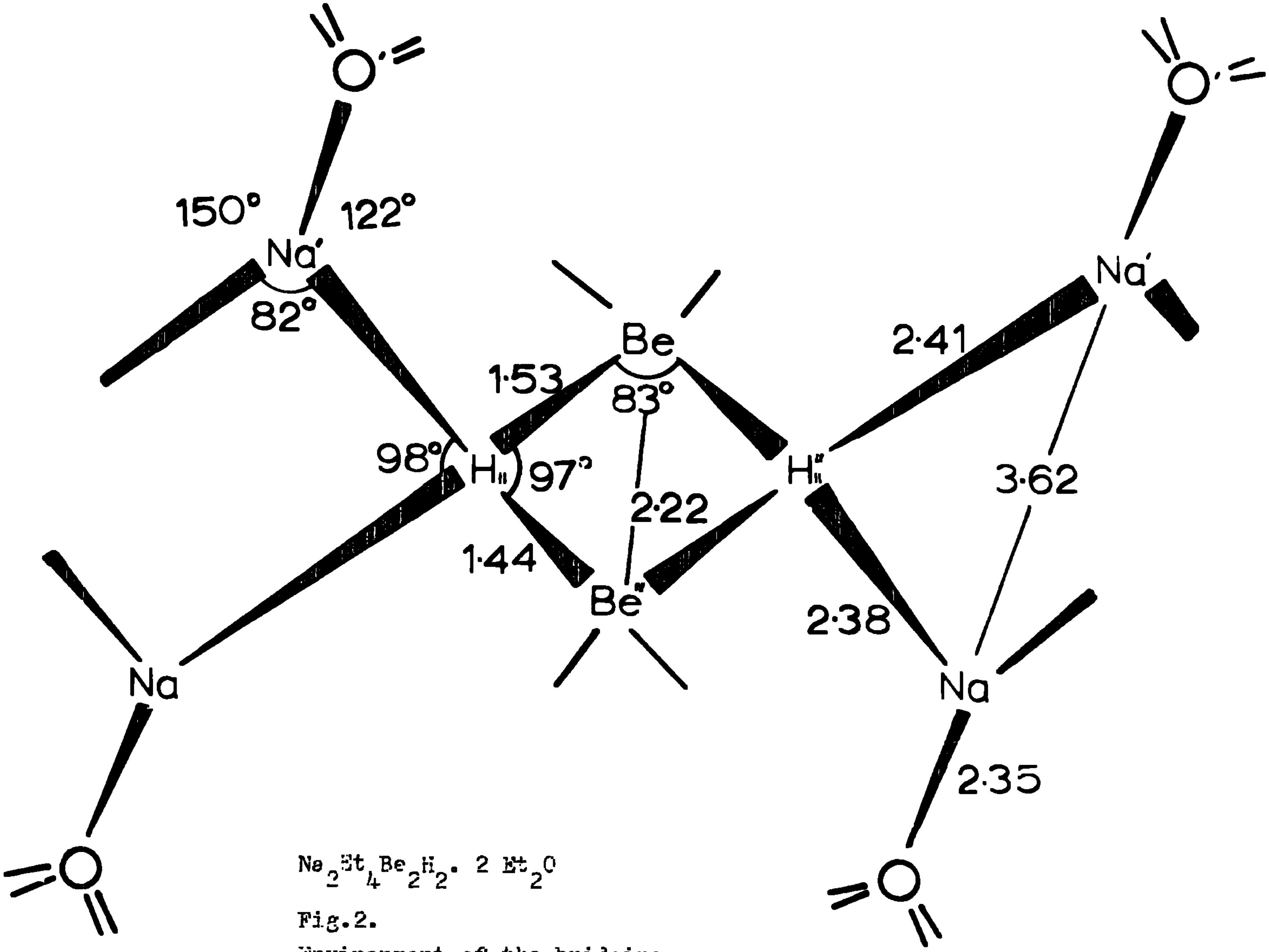
TABLE 5 (cont.)

| | | |
|---|---|---|
| C3-C4-H9 | 117 | |
| C3-C4-H10 | 102 | |
| H8-C4-H9 | 119 | |
| H8-C4-H10 | 106 | |
| H9-C4-H10 | 107 | |
| O-C6a-C5a | 114•3 | 3•7 |
| O-C6b-C5b | 105•7 | 2•5 |
| O-C7a-C8a | 108•0 | 1•9 |
| O-C7b-C8b | 119•9 | 4•5 |
| C2-Be-C3 | 118•0 | 0•4 |
| C2-Be-Be" | 123•9 | 0•4 |
| C3-Be-Be" | 118•1 | 0•4 |
| H11-Be-H11' | 83•4 | 1•7 |
| Na-H11-Na' | 98•1 | 1•1 |
| Na-H11-Be | 110•1 | 1•5 |
| Na-H11-Be" | 103•2 | 1•8 |
| Na'-H11-Be | 108•9 | 1•5 |
| Na'-H11-Be" | 136•3 | 2•0 |
| Be-H11-Be" | 96•6 | 1•8 |


H
H
C4
H
1·532
H
C3
H
O
H
1·810
2·350
H
C1
Na
H
H
1·542
1·766
Be
2·38
C2
1·53
H''11
H
H11
H
241
1·44
Be''
C''2
116·0°
H
H
118·0°
Na'
C''1
H
H
H
C''3
114·6°
O'
H
H
C''4
H
H

$Na_2Et_4Be_2H_2.2Et_2O$

Fig.1.


O
150°
122°
Na'
82°
Be
1·53
83°
98°
H
97°
2·22
1·44
Be"
Na
O
2·41
H"
Na'
O
3·62
2·38
Na
2·35
O

$Na_2Et_4Be_2H_2$. 2 $Et_2O$

Fig.2.

Environment of the bridging hydrogen atom.

The Na - H11 and Na' - H11 distances are 2.38± 0.03 and 2.41 ± 0.03 Å respectively and do not differ significantly from each other. The mean value of 2.40± 0.02 Å does not differ significantly from the Na - H distance of 2.440± 0.001 in sodium hydride (Brewer and Mastick, 1951). Likewise the Be- H11 and Be'' - H11 distances of 1.53± 0.03 and 1.44± 0.03 Å respectively are the same within experimental error.

The tetrahedral covalent radius of beryllium is 0.18 Å greater than that of boron, (Pauling, 1960). The length of the bridging boron-hydrogen bond as determined by electron diffraction in the gas phase (Hedberg and Schomaker, 1951) is 1.33 Å. From an x-ray structure analysis of β-diborane (Smith and Lipscomb, 1965) bridging B - H bond lenghts of 1.23± 0.02 and 1.25± 0.02 Å were obtained. If 0.18 Å is added to these values for a bridging B - H bond lengths then the possible values for the length of a Be - H bridging bond are 1.42Å and 1.51 Å. The value of 1.49± 0.02 Å obtained in this analysis does not differ significantly from either of these values. In $Be(BH_4)_2$ there is an unsymmetrical hydrogen bridge and infrared (Price,1949) and electron diffraction (Bauer , 1950) studies give values for the Be - H distance of 1.43 and 1.63 Å respectively. No. e.s.d.'s are quoted for these values. The shortness of bond lengths determined by x-ray methods, as compared with electron diffraction is apparently a general phenomenon (Lipscomb, 1954). In the case of $Na_2Et_4Be_2H_2 2Et_2O$ any lengthening of the Be-H11 bond lengths would result in shortening of the Na-H bond lengths.

The H-Be-H' and H-Na-H'' angles are 83.4±1.7° and 81.9±1.1° respectively and are not significantly different from each other. Likewise the Be-H-Be'' and Na-H-Na' angles of 96.6±1.8° and 98.1±1.1° are the same within experimental error.

The bond lengths of C2 - Be (1.760 ± 0.008 Å) and C3 - Be (1.810 ± 0.008 Å) differ significantly from one another, and no satisfactory reason can be given for this observation. The environments of C2 and C3 with respect to the sodium ions are different. The bond lengths are both shorter than the sum of the tetrahedral covalent radii of beryllium and carbon, which is 1.84 Å (Pauling, 1960). The bond lengths of C1 - C2 (1.542 ± 0.008 Å) and C3- C4 (1.532 ± 0.008 Å) do not differ from each other or from the value of 1.5445 Å found in diamond. The angles C1 - C2 - Be (116.8 ± 0.4°) and C4 - C3 - Be (114.6 ± 0.4°) are slightly larger than the tetrahedral angle as would be expected from a consideration of the relative sizes of the atoms bonded to C2 and C3. The angles C2 - Be - C3 (118.0 ± 0.4°), C2 - Be - $Be''$ (123.9 ± 0.4°) and C3 - Be - $Be''$ (118.1 ± 0.4°) are all near 120°, as are the corresponding angles in diborane.

The atoms, C2, C3, Be, $Be''$, $C2''$, $C3''$ are coplanar within experimental error. The equation of the mean plane is

$$-0.9957x' + 0.0919y + -0.0146z' -2.4556 = 0$$

The equation is referred to orthogonal axes where c' is perpendicular to a and b. The largest deviation from the plane is 0.002 Å.

The C - H bond lengths in the ethyl groups which are bonded to the beryllium atoms lie between 0.99 and 1.08 Å., compared with a value of 1.094 Å in methane (T.I.D. 1965).

The positions of the hydrogen atoms were determined from a Fo - Fc synthesis and no significance is attached to the different values obtained for the C - H bond length.

One ether molecule is bonded to each sodium ion by means of its oxygen atom. The Na - O distance of 2.350± 0.005 Å is equal to the sum of the ionic radii of sodium and oxygen (Pauling, 1960). This distance is similar to the value of 2.38 Å found in NaOH (Ernst, 1948). The atoms Na, Na', O, O', H11, H11' are nearly coplanar, the equation of the mean plane through these atoms being

$$-0.2364x' + 0.7063y - 0.6672z' = 0$$

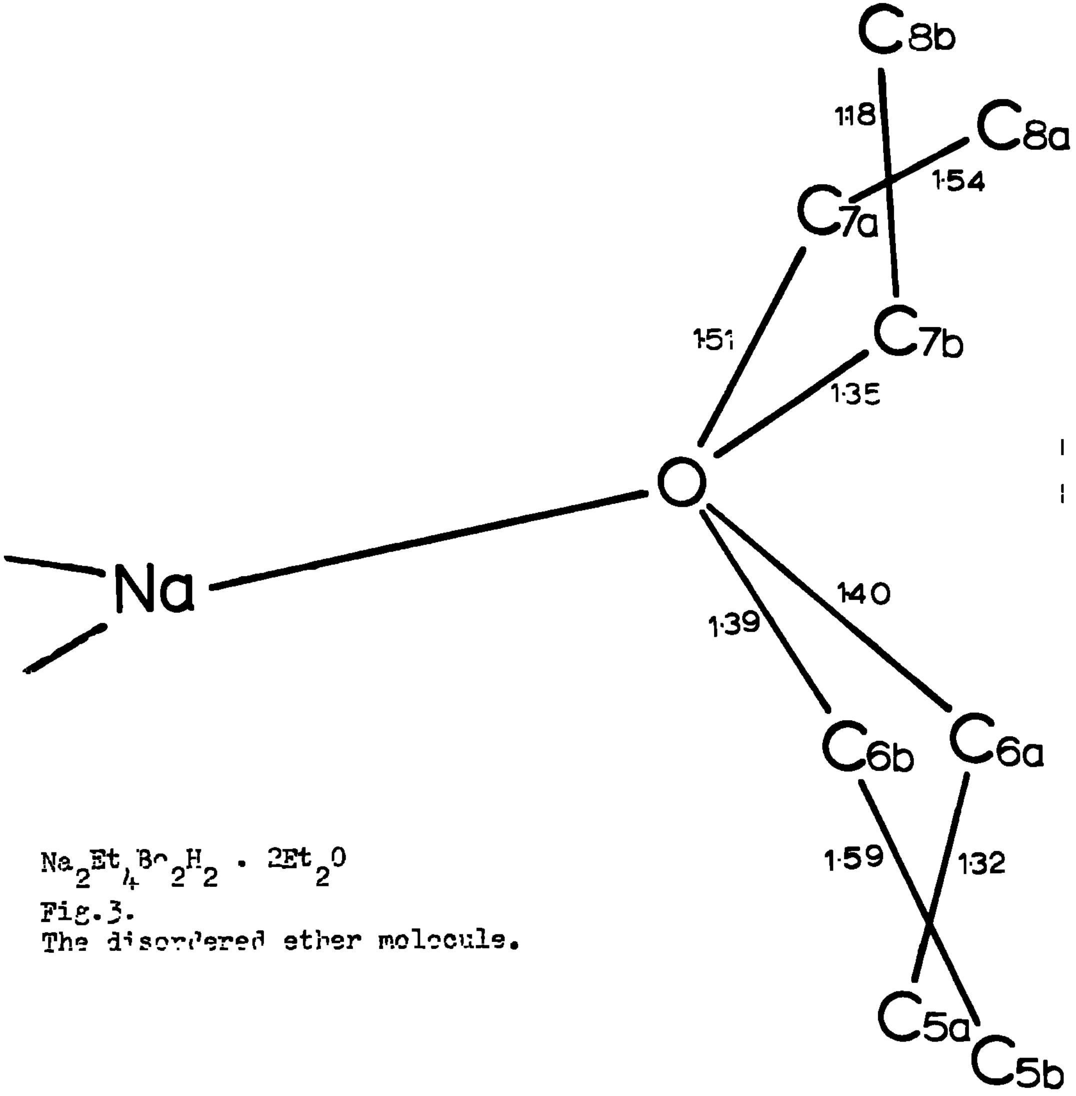
The deviations of the atoms from the plane are

| | |
|---|---|
| Na | +0.07 Å |
| H11 | -0.01 |
| O | -0.03 |

A drawing of the disordered ether molecule, giving the bond lengths is shown in Fig.3. In the disordered ether molecule the four carbon-oxygen distances range from 1.350± 0.034Å to 1.514±0.031 Å, none of which is considered significantly different from the value of 1.43± 0.03 Å found in $Me_2O$ (Schomaker, 1951). The mean C-O bond length is 1.41 Å. The bond lengths C5b-C6b (1.59±0.06 Å) and C7a-C8a (1.54±0.04 Å) are the same within experimental error as that found in diamond. The lengths C5a-C6a (1.32±0.06 Å) and C7b-C8b (1.18±0.05 Å) deviate significantly from the accepted value for a C-C single bond. The bond angles in the ether molecule do not differ greatly from those which might be expected.

The sums of the C-O-C and C-O-Na angles at oxygen in respect of the two sets of positions for the ether molecule are $358^{o}$and $360^{o}$. In both cases the oxygen atom lies almost in the plane of the three atoms to which it is bonded. In $EtMgBr.\ 2Et_2O$ (Guggenheimer and Rundle, 1964) there is a similar arrangement of atoms around the oxygen. In $PhMgBr.\ 2Et_2O$ (Stucky and Rundle, 1964) however the two carbon and one magnesium atom bonded to oxygen lie at three of the corners of a distorted tetrahedron. In the compounds in which the oxygen atom is coplanar with its three near neighbours, the bonding is said to be more ionic than that in which the oxygen has a tetrahedral environment (Guggenheimer and Rundle, 1964; Stucky and Rundle, 1964b).

It would seem that the disordered model used in the refinement, although a better representation than the ordered one, does not lead to a reliable set of molecular parameters. In the disordered ether molecule four non-bonding carbon-carbon distances of less that 1 Å are found and the temperature parameters are large.


C8b
1·18
C8a
1·54
C7a
C7b
1·51
1·35
O
Na
1·39
1·40
C6b
C6a
1·59
1·32
C5a
C5b

$Na_2Et_4Be_2H_2 \cdot 2Et_2O$

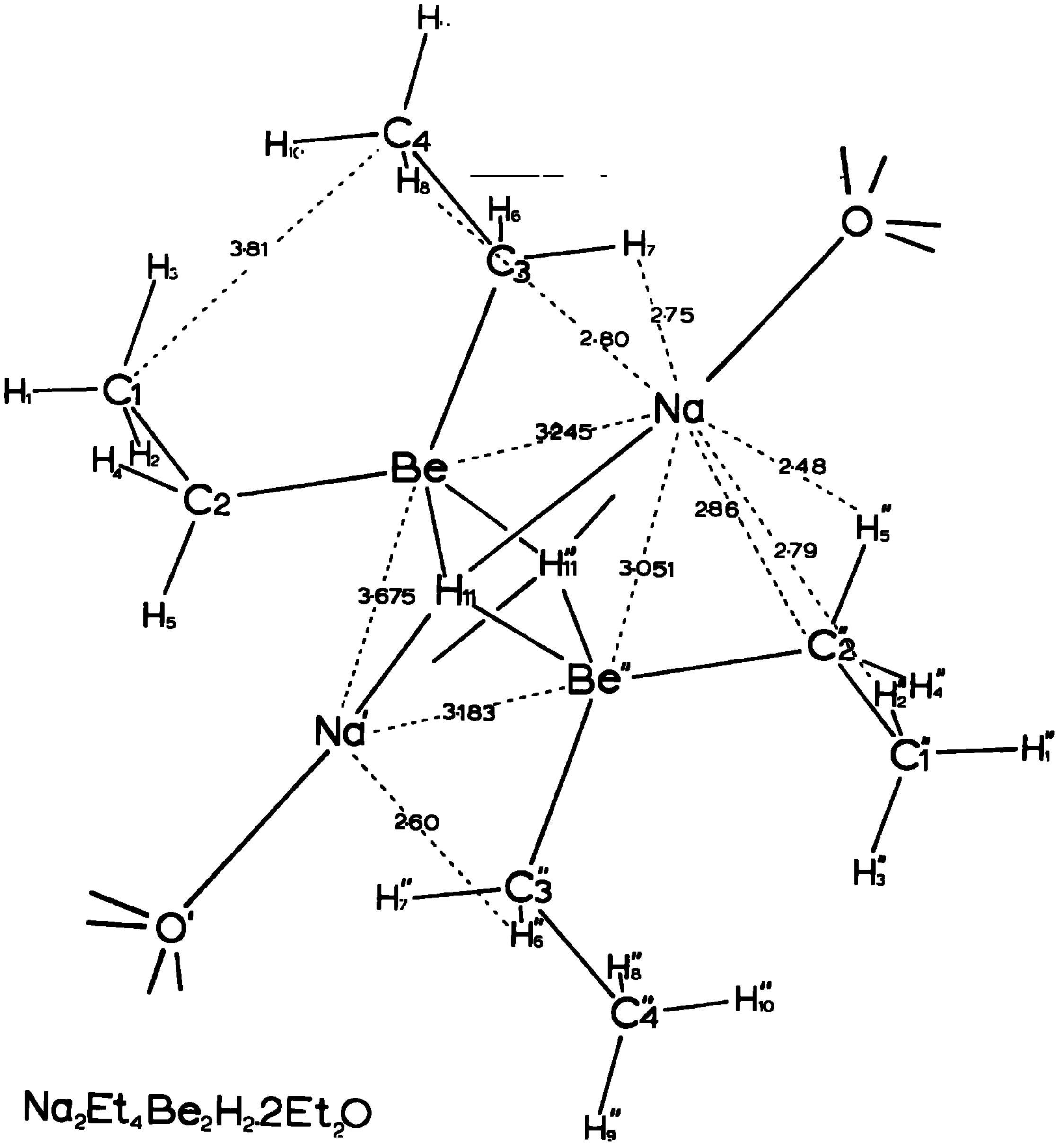
Fig.3.
The disordered ether molecule.

There is thus considerable overlap of the electron densities due to the disordered atoms.

The correlation coefficients between the parameters of different disordered carbon atoms are large and thus the covariance term in the expression for calculating bond length e.s.d.'s will be large (Cruickshank and Robertson, 1953). No allowance was made for this covariance in calculating the e.s.d.'s and they will thus be underestimated. In addition the thermal parameters of the disordered carbon atoms are large and so some apparent shortening of the C-C bonds would be expected due to the molecular librations. This effect would be greatest for the C-C bonds in the ether molecule, and the mean value of the C-C bond length in the ether molecule is found to be 1.41 Å.

In view of the high dissociation pressure of the ether in this complex, the possibility of ether loss must be considered. The temperature parameters of the oxygen atom are not appreciably greater than those of the other ordered atoms, suggesting that any such loss must be small.

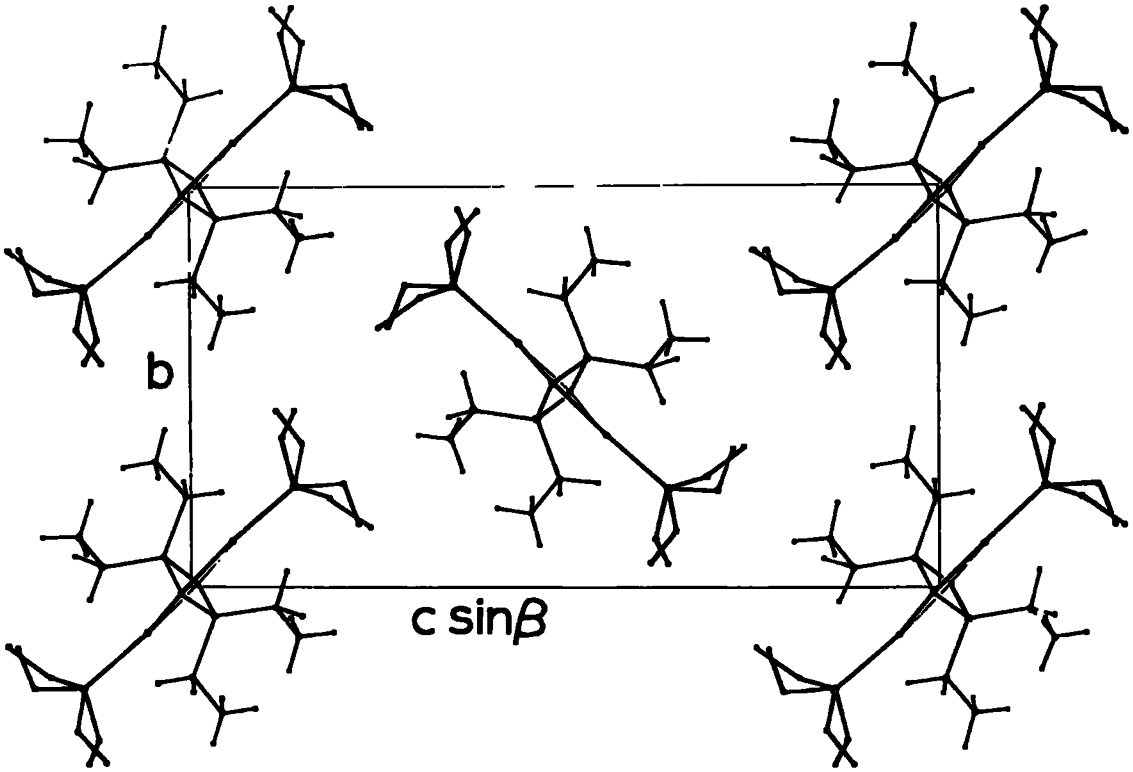
Some non-bonding distances are shown in Table 6 and Fig. 4. and the arrangement in the unit cell is shown in Fig. 5. If the value of the van der Waals radius of an alkyl hydrogen atom is taken as 1.2 Å, and those of a methylene or methyl carbon atom as being 2.0 Å (Pauling, 1960), then none of the van der Waals distances are unusually short. There are six alkyl hydrogen-sodium contacts ranging from 2.48 to 2.97 Å and two carbon-sodium contacts


H₉
H₁₀
C4
H₈
H₆
C3
H₇
O
3·81
H₃
2·75
2·80
H₁
C1
Na
3·245
H₄
H₂
Be
C2
2·48
2·86
H₅''
H₁₁''
3·051
2·79
3·675
H₁₁
H₅
C₂''
H₂''
H₄''
Be''
3·183
Na'
C₁''
H₁''
2·60
H₃''
H₇''
C₃''
H₆''
O'
H₈''
H₁₀''
C₄''
H₉''

$Na_2Et_4Be_2H_2{\cdot}2Et_2O$

Fig.4.

Non-bonding distances.



$Na_2Et_4Be_2H_2.2Et_2O$ [100] projection

Fig.5.

## $Na_2Et_4Be_2H_2 \cdot 2Et_2O$. TABLE 6

### Non-bonding distances (Å)

The position of the second atom is indicated by the operations given in column 2.

| | | |
|---|---|---|
| Na-Be | | 3·245 |
| Na-Be" | | 3·051 |
| Na'-Be | | 3·675 |
| Na'-Be" | | 3·183 |
| Na-H5" | | 2·49 |
| Na-H6 | (-1, 0, 0) | 2·60 |
| Na-H7 | | 2·75 |
| Na-H2" | | 2·79 |
| Na-H8 | | 2·80 |
| Na-C2' | | 2·86 |
| Na-H7 | (-1, 0, 0) | 2·97 |
| Na-C3 | (-1, 0, 0) | 3·02 |
| Na-C3' | | 3·13 |
| C1-C4 | | 3·81 |
| C1-C8 | $(1-x, -\frac{1}{2}+y, -\frac{1}{2}-z)$ | 3·84 |
| C1-C7 | $(1-x, -\frac{1}{2}+y, -\frac{1}{2}-z)$ | 3·95 |
| C1-C8 | $(x, \frac{1}{2}-y, \frac{1}{2}+z)$ | 3·97 |
| C3-C9 | (-1, 0, 0) | 3·71 |
| C3-O | (-1, 0, 0) | 3·79 |
| C4-C11 | $(-x, -\frac{1}{2}+y, -\frac{1}{2}-z)$ | 3·74 |
| C4-C10 | $(1-x, -\frac{1}{2}+y, -\frac{1}{2}-z)$ | 3·88 |
| C4-C11 | $(-x, -\frac{1}{2}+y, -\frac{1}{2}-z)$ | 3·74 |
| C4-C11 | | 3·88 |

of 2.86 and 3.02 Å. Five of the shorter non-bonding distances are for contacts between sodium and C3, or hydrogen atoms bonded to it, whereas there are only two comparable contacts between sodium and C2 or the hydrogen atoms bonded to it. This may be related to the observed difference in the Be-C bond lengths.

The Allred-Rochow electronegativities of beryllium and carbon are 1.47 and 2.50 respectively. Thus it would seem reasonable that in a $(Et_4Be_2H_2)^{2-}$ ion, the negative charge would be associated with the bridging hydrogen atoms and the ethyl groups, rather than with the beryllium atoms. The sodium ions have an ether oxygen atom (2.35 Å) and two bridging hydrogen atoms (2.38 and 2.41 Å) as near neighbours and six alkyl hydrogen atoms at distances of from 2.48 and 2.97 Å, two carbon atoms at 2.86 and 3.02 Å, and one sodium ion at 3.620 Å. Apart from the neighbouring sodium ion all of the atoms around a sodium ion would be expected to carry some negative charge, with which it can interact by ion-dipole forces.

## Discussion

The simplest description of the structure of the ether complex of sodium hydridodiethylberyllate is in terms of discrete $(NaOEt_2)^+$ and $(Et_4Be_2H_2)^{2-}$ ions. The C2-Be-Be'', C3-Be-Be'' and C2-Be-C3 angles are very close to 120° as are the corresponding angles in diborane, and it seems reasonable to describe the $BeH_2Be$ bridge in terms of three-centre molecular orbitals, as in the case of diborane. In diborane it has been calculated (Hamilton, 1956. Yamazaki, 1957) that the bridging hydrogen atoms carry a negative charge of 0.22 e. The electronegativities of boron and beryllium are 2.01 and 1.47 respectively. Because of the more electropositive nature of beryllium and the overall negative charge which the $\left[Et_4Be_2H_2\right]^{2-}$ ion carries, it would be expected that the bridging hydrogen atoms in this compound would carry a greater negative charge than those in $B_2H_6$.

The presence of a large negative charge on the bridging hydrogen atoms is consistent with the observed Na-H distances, which are the same as that in sodium hydride. The Be-H bonds would thus be expected to be highly polarised and it has been suggested (Gibb, 1962) that the Na-H interaction in sodium hydride is of mixed character.

The angles Na-H11-Na' and Be-H11-Be'' are the same within experimental error, as are the H11-Na-H11' and H11-Be-H11'' angles. The Na-Na' distance is shorter than that in metallic sodium and the Be-Be'' distance is equal to that in metallic beryllium. The similarity in environment of H11 with respect to the berylliums and sodiums suggest that the Na-H11 and Be-H11 interactions may not be too different in character.

$Na_2Et_4Be_2H_2.2Et_2O$.

Table 7.

Final Values of the Observed and Calculated Structure Factors.

| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
| 0 | 0 | 2 | 29,4 | 44,3 |
| 0 | 0 | 4 | 28,4 | -30,1 |
| 0 | 0 | 6 | 20,2 | 23,3 |
| 0 | 0 | 8 | 25,8 | 21,4 |
| 0 | 0 | 10 | 33,8 | -33,5 |
| 0 | 0 | 12 | 14,5 | -16,0 |
| 0 | 0 | 14 | 11,2 | 10,2 |
| 0 | 0 | 16 | 6,4 | 6,2 |
| 0 | 0 | 18 | 1,7 | 2,0 |
| 0 | 0 | 20 | 3,6 | 3,6 |
| 0 | 1 | 1 | 39,2 | 43,8 |
| 0 | 1 | 2 | 44,8 | -33,6 |
| 0 | 1 | 3 | 12,0 | 6,8 |
| 0 | 1 | 4 | 30,7 | 28,6 |
| 0 | 1 | 5 | 3,3 | 2,6 |
| 0 | 1 | 6 | 1,2 | -1,0 |
| 0 | 1 | 7 | 9,1 | -8,3 |
| 0 | 1 | 8 | 8,6 | 7,8 |
| 0 | 1 | 9 | 9,8 | -9,7 |
| 0 | 1 | 10 | 9,3 | 7,9 |
| 0 | 1 | 11 | 10,8 | -9,8 |
| 0 | 1 | 12 | 20,4 | 17,7 |
| 0 | 1 | 13 | 1,4 | 1,0 |
| 0 | 1 | 14 | 7,4 | 6,0 |
| 0 | 1 | 15 | 5,0 | 3,8 |
| 0 | 1 | 16 | < 1,6 | -1,8 |
| 0 | 1 | 17 | 4,3 | 4,0 |
| 0 | 2 | 0 | 50,4 | -48,4 |
| 0 | 2 | 1 | 18,8 | 18,1 |
| 0 | 2 | 2 | 5,7 | 2,4 |
| 0 | 2 | 3 | 18,8 | 9,5 |
| 0 | 2 | 4 | 49,2 | -34,9 |
| 0 | 2 | 5 | 97,0 | -83,7 |
| 0 | 2 | 6 | 26,8 | -24,7 |
| 0 | 2 | 7 | 16,9 | -18,6 |
| 0 | 2 | 8 | < 1,1 | 0,5 |
| 0 | 2 | 9 | 16,9 | 16,3 |
| 0 | 2 | 10 | 7,1 | 6,3 |
| 0 | 2 | 11 | 19,2 | 17,7 |
| 0 | 2 | 12 | 8,5 | -4,8 |
| 0 | 2 | 13 | 6,9 | 5,5 |
| 0 | 2 | 14 | 9,3 | -9,1 |
| 0 | 2 | 15 | 7,6 | 7,3 |
| 0 | 2 | 16 | < 1,6 | 1,2 |
| 0 | 2 | 17 | < 1,7 | 0,6 |
| 0 | 2 | 18 | < 1,7 | 1,8 |
| 0 | 2 | 19 | 2,8 | -2,4 |
| 0 | 2 | 20 | 4,4 | -3,8 |
| 0 | 3 | 1 | 2,8 | -2,9 |
| 0 | 3 | 2 | 12,5 | 13,3 |
| 0 | 3 | 3 | 3,7 | 2,4 |
| 0 | 3 | 4 | 47,7 | -42,6 |
| 0 | 3 | 5 | 6,5 | -6,5 |
| 0 | 3 | 6 | 21,8 | -19,7 |
| 0 | 3 | 7 | 7,8 | 8,9 |
| 0 | 3 | 8 | 14,2 | 11,7 |
| 0 | 3 | 9 | 8,5 | 7,8 |
| 0 | 3 | 10 | 16,0 | 14,1 |
| 0 | 3 | 11 | < 1,4 | 1,3 |
| 0 | 3 | 12 | 2,9 | 2,4 |
| 0 | 3 | 13 | 8,6 | -8,0 |
| 0 | 3 | 14 | 2,9 | 3,4 |
| 0 | 3 | 15 | 3,9 | -3,4 |
| 0 | 3 | 16 | 3,2 | 3,5 |
| 0 | 3 | 17 | 3,4 | -2,5 |
| 0 | 3 | 18 | 4,9 | -4,5 |
| 0 | 3 | 19 | 2,4 | -2,0 |
| 0 | 3 | 20 | 3,2 | -3,0 |
| 0 | 4 | 0 | 17,2 | -11,4 |
| 0 | 4 | 1 | 35,9 | 24,5 |
| 0 | 4 | 2 | 2,8 | -3,5 |
| 0 | 4 | 3 | 19,1 | 14,9 |
| 0 | 4 | 4 | 2,1 | 0,8 |
| 0 | 4 | 5 | 12,2 | 9,6 |
| 0 | 4 | 6 | 30,7 | 26,0 |
| 0 | 4 | 7 | < 1,2 | -0,6 |
| 0 | 4 | 8 | 26,0 | 22,9 |
| 0 | 4 | 9 | 13,1 | 12,7 |
| 0 | 4 | 10 | 20,3 | 18,5 |
| 0 | 4 | 11 | 9,5 | 8,5 |
| 0 | 4 | 12 | 2,6 | 3,0 |
| 0 | 4 | 13 | 2,3 | 2,6 |
| 0 | 4 | 14 | 1,6 | 2,3 |
| 0 | 4 | 15 | 6,0 | -6,5 |
| 0 | 4 | 16 | 3,2 | -2,4 |
| 0 | 4 | 17 | 4,4 | -3,9 |
| 0 | 4 | 18 | 1,7 | -1,9 |
| 0 | 5 | 1 | 30,1 | -22,7 |
| 0 | 5 | 2 | 6,8 | -4,4 |
| 0 | 5 | 3 | 2,3 | -2,2 |
| 0 | 5 | 4 | 23,4 | 20,3 |
| 0 | 5 | 5 | 10,3 | 9,0 |
| 0 | 5 | 6 | 14,2 | 14,6 |
| 0 | 5 | 7 | 23,1 | 14,4 |
| 0 | 5 | 8 | 4,1 | -3,7 |
| 0 | 5 | 9 | 22,0 | 18,5 |
| 0 | 5 | 10 | 1,8 | 1,2 |
| 0 | 5 | 11 | 16,7 | 13,5 |
| 0 | 5 | 12 | 3,5 | 3,8 |
| 0 | 5 | 13 | < 1,6 | 1,4 |
| 0 | 5 | 14 | 4,1 | -4,2 |
| 0 | 5 | 15 | 1,7 | -2,0 |
| 0 | 5 | 16 | 6,2 | -6,2 |
| 0 | 5 | 17 | < 1,7 | 0,6 |
| 0 | 5 | 18 | 3,2 | 2,8 |
| 0 | 5 | 19 | < 1,6 | 0,2 |
| 0 | 5 | 20 | 2,3 | 1,7 |
| 0 | 6 | 0 | 27,9 | -19,5 |
| 0 | 6 | 1 | 8,4 | 7,5 |
| 0 | 6 | 2 | 8,4 | -7,4 |
| 0 | 6 | 3 | 17,5 | 15,3 |
| 0 | 6 | 4 | 13,8 | 11,2 |
| 0 | 6 | 5 | 15,3 | 13,8 |
| 0 | 6 | 6 | 7,6 | -6,9 |
| 0 | 6 | 7 | 2,0 | -1,9 |
| 0 | 6 | 8 | 2,8 | -2,9 |
| 0 | 6 | 9 | < 1,5 | -0,3 |
| 0 | 6 | 10 | < 1,6 | -0,2 |
| 0 | 6 | 11 | < 1,6 | -0,1 |
| 0 | 6 | 12 | < 1,6 | 0,1 |
| 0 | 6 | 13 | 5,9 | -5,8 |
| 0 | 6 | 14 | 4,9 | -5,1 |
| 0 | 6 | 15 | 4,3 | -4,6 |
| 0 | 6 | 16 | < 1,7 | -0,1 |
| 0 | 6 | 17 | < 1,7 | 1,1 |
| 0 | 6 | 18 | < 1,6 | 0,5 |
| 0 | 6 | 19 | 2,5 | 2,6 |
| 0 | 7 | 1 | < 1,4 | 1,4 |
| 0 | 7 | 2 | 21,9 | 17,1 |
| 0 | 7 | 3 | 2,5 | -2,5 |
| 0 | 7 | 4 | 16,0 | 12,1 |
| 0 | 7 | 5 | 4,9 | -4,0 |
| 0 | 7 | 6 | 5,0 | 4,6 |
| 0 | 7 | 7 | 6,7 | -6,2 |
| 0 | 7 | 8 | < 1,6 | 2,4 |
| 0 | 7 | 9 | 6,3 | -5,3 |
| 0 | 7 | 10 | < 1,6 | -1,3 |
| 0 | 7 | 11 | 7,0 | -6,3 |
| 0 | 7 | 12 | 6,0 | -6,0 |
| 0 | 7 | 13 | < 1,7 | 0,0 |
| 0 | 7 | 14 | 4,9 | -4,6 |
| 0 | 7 | 15 | 1,8 | 1,1 |
| 0 | 8 | 0 | 11,4 | 10,6 |
| 0 | 8 | 1 | < 1,6 | -1,0 |
| 0 | 8 | 2 | < 1,6 | 0,9 |
| 0 | 8 | 3 | 4,0 | 4,0 |
| 0 | 8 | 4 | 2,8 | -2,6 |
| 0 | 8 | 5 | < 1,6 | -1,1 |
| 0 | 8 | 6 | 2,6 | 2,8 |
| 0 | 8 | 7 | < 1,6 | -0,9 |
| 0 | 8 | 8 | 3,2 | -3,1 |
| 0 | 8 | 9 | 4,8 | -5,1 |
| 0 | 8 | 10 | 6,4 | -5,5 |
| 0 | 8 | 11 | 2,8 | -2,6 |
| 0 | 8 | 12 | 2,5 | 2,6 |
| 0 | 8 | 13 | < 1,7 | -0,9 |
| 0 | 8 | 14 | 6,1 | 6,3 |
| 0 | 8 | 15 | < 1,6 | -0,5 |
| 0 | 8 | 16 | 1,6 | 1,3 |
| 0 | 9 | 1 | 6,7 | 6,4 |
| 0 | 9 | 2 | 7,9 | -6,4 |
| 0 | 9 | 3 | 7,5 | 7,2 |
| 0 | 9 | 4 | 4,5 | -3,3 |
| 0 | 9 | 5 | 2,7 | 3,1 |
| 0 | 9 | 6 | 5,1 | -5,2 |
| 0 | 9 | 7 | 2,7 | -3,0 |
| 0 | 9 | 8 | 7,4 | -6,1 |
| 0 | 9 | 9 | 4,6 | -4,0 |
| 0 | 9 | 10 | 2,1 | -2,1 |
| 0 | 9 | 11 | 1,7 | 2,1 |
| 0 | 9 | 12 | 2,1 | 2,2 |
| 0 | 9 | 13 | 3,3 | 3,5 |
| 0 | 9 | 14 | < 1,5 | 0,9 |
| 0 | 9 | 15 | 2,1 | 1,9 |
| 0 | 10 | 0 | 1,7 | -1,7 |
| 0 | 10 | 1 | < 1,7 | 1,3 |
| 0 | 10 | 2 | 3,3 | 3,5 |
| 0 | 10 | 3 | 2,8 | -2,5 |
| 0 | 10 | 4 | 2,8 | 2,5 |
| 0 | 10 | 5 | 6,0 | -5,8 |
| 0 | 10 | 6 | 2,5 | -1,9 |
| 0 | 10 | 7 | 3,2 | -2,8 |
| 0 | 10 | 8 | 4,2 | -3,4 |
| 0 | 10 | 9 | 2,5 | 1,5 |
| 0 | 11 | 1 | 3,2 | -3,2 |
| 0 | 11 | 2 | 2,4 | -1,7 |
| 0 | 11 | 3 | 3,8 | -2,7 |
| 0 | 11 | 4 | 4,1 | -4,0 |
| 0 | 12 | 0 | 1,9 | -1,7 |
| 0 | 12 | 1 | 1,6 | -1,3 |
| 0 | 12 | 2 | < 1,6 | -1,0 |
| 0 | 12 | 3 | 2,2 | -2,6 |
| 1 | 0 | -20 | 2,8 | 2,8 |
| 1 | 0 | -18 | 1,4 | -1,6 |
| 1 | 0 | -16 | 9,7 | 8,3 |
| 1 | 0 | -14 | 9,2 | 8,9 |
| 1 | 0 | -12 | 19,8 | -19,9 |
| 1 | 0 | -10 | 16,0 | -16,4 |
| 1 | 0 | -8 | 19,0 | -16,2 |
| 1 | 0 | -6 | 64,0 | -55,5 |
| 1 | 0 | -4 | 40,0 | -44,0 |
| 1 | 0 | -2 | 18,9 | 19,2 |
| 1 | 0 | 0 | --- | 18,9 |
| 1 | 0 | 2 | 103,8 | -104,8 |
| 1 | 0 | 4 | 3,0 | 2,6 |
| 1 | 0 | 6 | 1,9 | 1,2 |
| 1 | 0 | 8 | 31,3 | -32,6 |
| 1 | 0 | 10 | 18,2 | -19,7 |
| 1 | 0 | 12 | 16,2 | 14,2 |
| 1 | 0 | 14 | 10,2 | 9,5 |
| 1 | 1 | -21 | 2,3 | 2,3 |
| 1 | 1 | -20 | < 1,5 | -0,4 |
| 1 | 1 | -19 | < 1,4 | 0,9 |
| 1 | 1 | -18 | < 1,4 | -0,7 |
| 1 | 1 | -17 | 1,3 | 1,5 |
| 1 | 1 | -16 | < 1,2 | 1,4 |
| 1 | 1 | -15 | 3,3 | 3,1 |
| 1 | 1 | -14 | 3,9 | -5,1 |
| 1 | 1 | -13 | 3,8 | 4,2 |
| 1 | 1 | -12 | 3,3 | -3,6 |
| 1 | 1 | -11 | 9,2 | -10,3 |
| 1 | 1 | -10 | 28,9 | 25,8 |
| 1 | 1 | -9 | 21,2 | -20,2 |
| 1 | 1 | -8 | 21,2 | 22,1 |
| 1 | 1 | -7 | 21,8 | -19,6 |
| 1 | 1 | -6 | 34,8 | 39,2 |
| 1 | 1 | -5 | 36,2 | 31,2 |
| 1 | 1 | -4 | 2,8 | 5,3 |
| 1 | 1 | -3 | 25,6 | 24,8 |
| 1 | 1 | -2 | 72,3 | 68,2 |
| 1 | 1 | -1 | 5,2 | -2,2 |
| 1 | 1 | 0 | 46,8 | -44,1 |
| 1 | 1 | 1 | 16,7 | -17,8 |
| 1 | 1 | 2 | 63,5 | -62,6 |
| 1 | 1 | 3 | 15,9 | 13,1 |
| 1 | 1 | 4 | 4,6 | -5,7 |
| 1 | 1 | 5 | 11,5 | -12,8 |
| 1 | 1 | 6 | < 0,9 | -1,5 |
| 1 | 1 | 7 | 35,1 | -32,8 |
| 1 | 1 | 8 | 3,9 | -3,6 |
| 1 | 1 | 9 | 20,0 | -20,2 |
| 1 | 1 | 10 | 8,8 | 8,6 |
| 1 | 1 | 11 | 4,3 | 4,7 |
| 1 | 1 | 12 | 13,8 | 13,0 |
| 1 | 1 | 13 | 10,7 | 10,2 |
| 1 | 1 | 14 | 3,1 | 4,1 |
| 1 | 1 | 15 | 3,8 | 4,8 |
| 1 | 1 | 16 | < 1,5 | -0,8 |
| 1 | 1 | 17 | 2,1 | 2,1 |
| 1 | 2 | -21 | 2,3 | 2,4 |
| 1 | 2 | -20 | < 1,4 | 0,7 |
| 1 | 2 | -19 | < 1,4 | 1,6 |
| 1 | 2 | -18 | 3,9 | 4,1 |
| 1 | 2 | -17 | 4,3 | -5,0 |
| 1 | 2 | -16 | 1,3 | -1,5 |
| 1 | 2 | -15 | 5,9 | -6,3 |
| 1 | 2 | -14 | < 1,2 | -1,6 |
| 1 | 2 | -13 | 7,3 | -6,8 |
| 1 | 2 | -12 | 19,9 | 16,7 |
| 1 | 2 | -11 | 12,6 | -12,7 |
| 1 | 2 | -10 | 7,6 | 8,5 |
| 1 | 2 | -9 | 8,4 | 8,6 |
| 1 | 2 | -8 | 10,9 | -9,3 |
| 1 | 2 | -7 | 37,5 | 36,2 |
| 1 | 2 | -6 | 2,9 | 3,2 |
| 1 | 2 | -5 | 32,6 | 29,1 |
| 1 | 2 | -4 | 62,0 | 47,4 |
| 1 | 2 | -3 | 18,0 | -12,9 |
| 1 | 2 | -2 | 61,5 | 34,9 |
| 1 | 2 | -1 | 54,9 | 40,6 |
| 1 | 2 | 0 | 28,8 | -28,5 |
| 1 | 2 | 1 | < 0,7 | 1,5 |
| 1 | 2 | 2 | 31,1 | 30,8 |
| 1 | 2 | 3 | 34,0 | -36,7 |
| 1 | 2 | 4 | 8,1 | 8,4 |
| 1 | 2 | 5 | 24,0 | -25,4 |
| 1 | 2 | 6 | 6,7 | -7,3 |
| 1 | 2 | 7 | 12,5 | 13,5 |
| 1 | 2 | 8 | 1,0 | -0,9 |
| 1 | 2 | 9 | 3,9 | 4,3 |
| 1 | 2 | 10 | 9,0 | 8,7 |
| 1 | 2 | 11 | 4,8 | 5,0 |
| 1 | 2 | 12 | 4,6 | 5,2 |
| 1 | 2 | 13 | 4,3 | 4,9 |
| 1 | 2 | 14 | 3,5 | 3,8 |
| 1 | 2 | 15 | < 1,4 | 2,6 |
| 1 | 2 | 16 | 4,1 | 4,6 |
| 1 | 2 | 17 | 3,3 | -3,5 |
| 1 | 2 | 18 | < 1,5 | 1,6 |
| 1 | 2 | 19 | 2,8 | -2,4 |
| 1 | 3 | -20 | 3,1 | 3,0 |
| 1 | 3 | -19 | < 1,4 | -0,1 |
| 1 | 3 | -18 | < 1,4 | -0,9 |
| 1 | 3 | -17 | 2,4 | -2,7 |
| 1 | 3 | -16 | 13,7 | -12,6 |
| 1 | 3 | -15 | 2,6 | 3,0 |
| 1 | 3 | -14 | 4,6 | -4,9 |
| 1 | 3 | -13 | 9,8 | 9,7 |
| 1 | 3 | -12 | 3,2 | -2,7 |
| 1 | 3 | -11 | 15,2 | 14,7 |
| 1 | 3 | -10 | 8,1 | -7,3 |
| 1 | 3 | -9 | 1,6 | -1,9 |
| 1 | 3 | -8 | < 0,9 | 0,9 |
| 1 | 3 | -7 | 10,4 | 10,7 |
| 1 | 3 | -6 | 23,5 | 22,0 |
| 1 | 3 | -5 | 7,1 | -9,0 |
| 1 | 3 | -4 | 29,6 | 26,9 |
| 1 | 3 | -3 | 21,8 | -20,7 |
| 1 | 3 | -2 | 1,9 | 0,5 |
| 1 | 3 | -1 | 50,3 | -41,3 |
| 1 | 3 | 0 | 1,3 | -1,1 |
| 1 | 3 | 1 | 0,8 | -0,8 |
| 1 | 3 | 2 | 34,6 | -28,9 |
| 1 | 3 | 3 | 7,4 | 8,2 |
| 1 | 3 | 4 | 46,1 | -41,0 |
| 1 | 3 | 5 | 22,8 | 21,3 |
| 1 | 3 | 6 | < 1,0 | 0,7 |
| 1 | 3 | 7 | 20,7 | 20,6 |
| 1 | 3 | 8 | 5,8 | 6,6 |
| 1 | 3 | 9 | 14,4 | 15,3 |
| 1 | 3 | 10 | 2,1 | -2,3 |
| 1 | 3 | 11 | 3,4 | 3,7 |
| 1 | 3 | 12 | < 1,3 | -0,8 |
| 1 | 3 | 13 | 2,4 | 2,0 |
| 1 | 3 | 14 | 5,0 | 6,2 |
| 1 | 3 | 15 | < 1,5 | -0,6 |
| 1 | 3 | 16 | < 1,5 | 0,9 |
| 1 | 3 | 17 | 2,9 | -3,5 |
| 1 | 3 | 18 | 3,0 | -3,9 |
| 1 | 3 | 19 | < 1,4 | -1,2 |
| 1 | 3 | 20 | 1,3 | -0,9 |
| 1 | 4 | -21 | 2,3 | -1,6 |
| 1 | 4 | -20 | < 1,3 | -0,9 |
| 1 | 4 | -19 | < 1,4 | 0,6 |
| 1 | 4 | -18 | 6,7 | -6,5 |
| 1 | 4 | -17 | < 1,4 | 0,2 |
| 1 | 4 | -16 | 6,6 | -6,5 |
| 1 | 4 | -15 | 3,0 | -3,9 |
| 1 | 4 | -14 | 1,8 | 1,6 |
| 1 | 4 | -13 | 4,3 | -4,7 |
| 1 | 4 | -12 | 3,1 | 3,0 |
| 1 | 4 | -11 | 8,4 | 8,3 |
| 1 | 4 | -10 | 5,7 | 6,0 |
| 1 | 4 | -9 | 8,5 | 9,8 |
| 1 | 4 | -8 | 3,0 | 2,4 |
| 1 | 4 | -7 | 6,0 | 6,2 |
| 1 | 4 | -6 | 4,8 | 4,7 |
| 1 | 4 | -5 | 1,7 | -1,2 |
| 1 | 4 | -4 | 29,2 | -23,7 |
| 1 | 4 | -3 | 12,6 | 13,2 |
| 1 | 4 | -2 | 39,8 | -35,5 |
| 1 | 4 | -1 | 3,4 | -2,8 |
| 1 | 4 | 0 | 26,8 | -23,7 |
| 1 | 4 | 1 | 16,3 | -13,5 |
| 1 | 4 | 2 | 16,5 | -15,9 |
| 1 | 4 | 3 | 31,0 | -25,8 |
| 1 | 4 | 4 | < 1,0 | -0,8 |
| 1 | 4 | 5 | 5,9 | -6,7 |
| 1 | 4 | 6 | 22,5 | 20,3 |
| 1 | 4 | 7 | 1,5 | -1,4 |
| 1 | 4 | 8 | 6,2 | 7,7 |
| 1 | 4 | 9 | < 1,7 | 0,2 |
| 1 | 4 | 10 | 2,5 | -2,4 |
| 1 | 4 | 11 | < 1,3 | -1,7 |
| 1 | 4 | 12 | 3,9 | -5,0 |
| 1 | 4 | 13 | 2,0 | 2,6 |
| 1 | 4 | 14 | 3,2 | -3,3 |
| 1 | 4 | 15 | 1,5 | 0,5 |
| 1 | 4 | 16 | 7,1 | -7,2 |
| 1 | 4 | 17 | < 1,5 | 0,3 |
| 1 | 4 | 18 | 3,8 | -3,6 |
| 1 | 5 | -19 | 1,4 | -1,8 |
| 1 | 5 | -18 | 2,4 | 2,5 |
| 1 | 5 | -17 | 5,0 | -5,5 |
| 1 | 5 | -16 | 6,2 | 5,8 |
| 1 | 5 | -15 | 10,5 | -9,0 |
| 1 | 5 | -14 | 2,3 | -3,1 |
| 1 | 5 | -13 | 2,9 | -3,0 |
| 1 | 5 | -12 | 5,3 | 5,1 |
| 1 | 5 | -11 | 3,2 | 3,8 |
| 1 | 5 | -10 | 8,6 | 8,9 |
| 1 | 5 | -9 | 2,5 | 3,1 |
| 1 | 5 | -8 | 6,4 | -5,9 |
| 1 | 5 | -7 | 3,6 | 3,5 |
| 1 | 5 | -6 | 29,5 | -24,2 |
| 1 | 5 | -5 | 4,3 | 3,9 |
| 1 | 5 | -4 | 8,7 | 9,6 |
| 1 | 5 | -3 | 13,9 | -13,0 |
| 1 | 5 | -2 | 24,2 | 19,4 |
| 1 | 5 | -1 | 19,2 | -16,9 |
| 1 | 5 | 0 | 1,0 | 0,3 |
| 1 | 5 | 1 | 5,1 | -6,2 |
| 1 | 5 | 2 | < 1,0 | 1,3 |
| 1 | 5 | 3 | 3,2 | -3,3 |
| 1 | 5 | 4 | 26,0 | 24,3 |
| 1 | 5 | 5 | < 1,1 | -0,3 |
| 1 | 5 | 6 | 4,8 | 5,6 |
| 1 | 5 | 7 | 4,4 | 5,4 |
| 1 | 5 | 8 | 4,1 | -5,6 |
| 1 | 5 | 9 | 1,8 | 1,6 |

| h | k | l | \|Fo\| | Fc | h | k | l | \|Fo\| | Fc | h | k | l | \|Fo\| | Fc | h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 5 | 10 | 5,5 | -6,9 | 1 | 8 | 8 | 6,6 | -7,9 | 2 | 1 | -9 | 3,5 | -4,0 | 2 | 4 | -13 | 1,5 | -2,2 |
| 1 | 5 | 11 | 3,9 | -3,8 | 1 | 8 | 9 | 2,5 | -3,3 | 2 | 1 | -8 | 2,9 | -2,9 | 2 | 4 | -12 | 4,2 | 4,3 |
| 1 | 5 | 12 | 3,7 | -4,4 | 1 | 8 | 10 | 6,0 | -5,7 | 2 | 1 | -7 | 1,7 | -2,0 | 2 | 4 | -11 | 7,6 | -7,0 |
| 1 | 5 | 13 | 8,0 | -8,0 | 1 | 8 | 11 | 1,5 | -1,2 | 2 | 1 | -6 | < 1,3 | 0,7 | 2 | 4 | -10 | 8,2 | 9,0 |
| 1 | 5 | 14 | 3,9 | -4,2 | 1 | 9 | -13 | 2,7 | -2,7 | 2 | 1 | -5 | 6,5 | 5,6 | 2 | 4 | -9 | 3,7 | -3,2 |
| 1 | 5 | 15 | 5,1 | -5,1 | 1 | 9 | -12 | 2,0 | -2,4 | 2 | 1 | -4 | 6,4 | 5,5 | 2 | 4 | -8 | 15,6 | 14,4 |
| 1 | 5 | 16 | < 1,5 | 0,1 | 1 | 9 | -11 | 3,9 | -4,0 | 2 | 1 | -3 | 6,2 | 6,3 | 2 | 4 | -7 | 1,0 | 1,5 |
| 1 | 5 | 17 | 1,7 | -1,8 | 1 | 9 | -10 | < 1,4 | 1,3 | 2 | 1 | -2 | 17,6 | 16,9 | 2 | 4 | -6 | 3,2 | -3,3 |
| 1 | 6 | -17 | 2,0 | 2,9 | 1 | 9 | -9 | 4,9 | -5,9 | 2 | 1 | -1 | 24,2 | 23,1 | 2 | 4 | -5 | 5,4 | 6,3 |
| 1 | 6 | -16 | < 1,4 | -1,4 | 1 | 9 | -8 | < 1,4 | 0,5 | 2 | 1 | 0 | 37,5 | -40,1 | 2 | 4 | -4 | 25,3 | -24,0 |
| 1 | 6 | -15 | 2,4 | 2,6 | 1 | 9 | -7 | 4,0 | -4,8 | 2 | 1 | 1 | 20,4 | 20,6 | 2 | 4 | -3 | 5,1 | 5,7 |
| 1 | 6 | -14 | 5,1 | -5,2 | 1 | 9 | -6 | 2,5 | -2,1 | 2 | 1 | 2 | 21,3 | -16,8 | 2 | 4 | -2 | 9,3 | -9,3 |
| 1 | 6 | -13 | 3,9 | 4,1 | 1 | 9 | -5 | 3,8 | -4,2 | 2 | 1 | 3 | < 1,0 | 1,0 | 2 | 4 | -1 | 1,1 | 1,4 |
| 1 | 6 | -12 | < 1,3 | 1,5 | 1 | 9 | -4 | < 1,4 | -0,9 | 2 | 1 | 4 | 3,7 | 4,9 | 2 | 4 | 0 | < 1,1 | 2,3 |
| 1 | 6 | -11 | 5,1 | 5,4 | 1 | 9 | -3 | 2,0 | 1,9 | 2 | 1 | 5 | 17,1 | -17,8 | 2 | 4 | 1 | 6,4 | 8,3 |
| 1 | 6 | -10 | 2,0 | 2,4 | 1 | 9 | -2 | 2,8 | 3,6 | 2 | 1 | 6 | 6,5 | -7,3 | 2 | 4 | 2 | 3,5 | -3,7 |
| 1 | 6 | -9 | < 1,2 | 0,4 | 1 | 9 | -1 | 2,0 | 2,6 | 2 | 1 | 7 | 7,9 | -7,3 | 2 | 4 | 3 | 8,6 | 9,2 |
| 1 | 6 | -8 | 1,7 | 1,6 | 1 | 9 | 0 | 2,0 | -2,4 | 2 | 1 | 8 | 3,4 | -3,7 | 2 | 4 | 4 | 12,7 | 14,9 |
| 1 | 6 | -7 | 17,0 | -14,1 | 1 | 9 | 1 | < 1,4 | 1,8 | 2 | 1 | 9 | 1,5 | 2,0 | 2 | 4 | 5 | 2,4 | 1,9 |
| 1 | 6 | -6 | 2,0 | 2,0 | 1 | 9 | 2 | < 1,4 | -1,1 | 2 | 1 | 10 | 10,2 | 9,8 | 2 | 4 | 6 | 22,0 | 21,6 |
| 1 | 6 | -5 | 19,2 | -15,4 | 1 | 9 | 3 | < 1,4 | -2,1 | 2 | 1 | 11 | 2,8 | 2,5 | 2 | 4 | 7 | < 1,5 | -0,7 |
| 1 | 6 | -4 | 11,3 | 10,4 | 1 | 9 | 4 | 3,2 | 2,9 | 2 | 1 | 12 | 4,5 | 4,5 | 2 | 4 | 8 | 5,4 | 6,2 |
| 1 | 6 | -3 | 3,3 | 2,3 | 1 | 9 | 5 | 4,9 | -5,2 | 2 | 1 | 13 | < 1,7 | -0,3 | 2 | 4 | 9 | 3,2 | 3,0 |
| 1 | 6 | -2 | 6,0 | -5,2 | 1 | 9 | 6 | 1,5 | 1,1 | 2 | 1 | 14 | 3,5 | -3,1 | 2 | 4 | 10 | < 1,6 | -1,4 |
| 1 | 6 | -1 | 10,0 | 10,0 | 1 | 9 | 7 | 5,3 | -6,2 | 2 | 1 | 15 | < 1,5 | 1,1 | 2 | 4 | 11 | 4,2 | 4,2 |
| 1 | 6 | 0 | 2,3 | -2,5 | 1 | 9 | 8 | < 1,5 | -0,4 | 2 | 1 | 16 | 3,1 | -2,3 | 2 | 4 | 12 | < 1,7 | -0,2 |
| 1 | 6 | 1 | 16,4 | 14,6 | 1 | 9 | 9 | 2,5 | -2,2 | 2 | 2 | -14 | 5,2 | -5,9 | 2 | 4 | 13 | < 1,7 | 0,1 |
| 1 | 6 | 2 | 13,1 | 11,2 | 1 | 9 | 10 | 2,0 | 1,6 | 2 | 2 | -13 | 8,1 | -7,7 | 2 | 4 | 14 | 2,2 | -2,2 |
| 1 | 6 | 3 | 17,3 | 18,7 | 1 | 9 | 11 | < 1,4 | -0,1 | 2 | 2 | -12 | < 1,9 | -0,4 | 2 | 4 | 15 | 3,5 | -3,2 |
| 1 | 6 | 4 | 15,8 | 13,5 | 1 | 9 | 12 | 1,9 | 2,0 | 2 | 2 | -11 | 4,6 | -5,0 | 2 | 4 | 16 | 2,4 | -2,8 |
| 1 | 6 | 5 | 10,6 | 10,7 | 1 | 9 | 13 | 1,3 | -1,0 | 2 | 2 | -10 | 11,7 | -11,8 | 2 | 5 | -16 | 4,5 | 3,5 |
| 1 | 6 | 6 | 2,5 | 2,6 | 1 | 10 | -9 | 1,4 | 0,5 | 2 | 2 | -9 | 12,5 | 13,6 | 2 | 5 | -15 | 1,5 | 2,2 |
| 1 | 6 | 7 | < 1,8 | 0,1 | 1 | 10 | -8 | 2,0 | -2,2 | 2 | 2 | -8 | 13,0 | -13,2 | 2 | 5 | -14 | < 1,6 | 0,3 |
| 1 | 6 | 8 | 6,6 | 6,9 | 1 | 10 | -7 | 4,7 | 5,4 | 2 | 2 | -7 | 19,8 | 20,4 | 2 | 5 | -13 | 8,7 | 8,3 |
| 1 | 6 | 9 | 3,6 | -4,1 | 1 | 10 | -6 | 2,0 | -2,4 | 2 | 2 | -6 | 9,5 | -9,7 | 2 | 5 | -12 | < 1,5 | 0,1 |
| 1 | 6 | 10 | 3,1 | 3,6 | 1 | 10 | -5 | 3,2 | 2,3 | 2 | 2 | -5 | 4,3 | -4,5 | 2 | 5 | -11 | 6,3 | 6,4 |
| 1 | 6 | 11 | 8,4 | -9,5 | 1 | 10 | -4 | 1,5 | -1,5 | 2 | 2 | -4 | 6,8 | -5,9 | 2 | 5 | -10 | 11,8 | -10,7 |
| 1 | 6 | 12 | 1,6 | -1,0 | 1 | 10 | -3 | < 1,5 | -1,1 | 2 | 2 | -3 | 17,2 | -16,2 | 2 | 5 | -9 | 5,4 | 6,1 |
| 1 | 6 | 13 | 6,4 | -7,0 | 1 | 10 | -2 | < 1,5 | -0,4 | 2 | 2 | -2 | 15,0 | -13,2 | 2 | 5 | -8 | 13,1 | -12,4 |
| 1 | 6 | 14 | 3,3 | -3,6 | 1 | 10 | -1 | 1,5 | -1,2 | 2 | 2 | -1 | 21,4 | -19,5 | 2 | 5 | -7 | 3,6 | 3,4 |
| 1 | 6 | 15 | < 1,6 | 0,7 | 1 | 10 | 0 | < 1,5 | 1,2 | 2 | 2 | 0 | < 0,9 | -1,3 | 2 | 5 | -6 | 7,7 | -7,2 |
| 1 | 6 | 16 | < 1,4 | 0,9 | 1 | 10 | 1 | < 1,6 | -0,8 | 2 | 2 | 1 | 37,9 | -34,6 | 2 | 5 | -5 | 8,2 | -7,5 |
| 1 | 6 | 17 | 2,0 | 1,8 | 1 | 10 | 2 | 2,9 | 3,2 | 2 | 2 | 2 | 16,2 | 16,5 | 2 | 5 | -4 | < 1,3 | -1,2 |
| 1 | 7 | -17 | 1,9 | 1,9 | 1 | 10 | 3 | 4,9 | -4,9 | 2 | 2 | 3 | 15,6 | -17,2 | 2 | 5 | -3 | 18,6 | -17,8 |
| 1 | 7 | -16 | < 1,4 | -1,6 | 1 | 10 | 4 | 2,1 | -2,4 | 2 | 2 | 4 | 9,8 | -11,5 | 2 | 5 | -2 | 10,5 | -12,4 |
| 1 | 7 | -15 | < 1,4 | 0,3 | 1 | 10 | 5 | 2,9 | -2,7 | 2 | 2 | 5 | < 1,2 | 1,9 | 2 | 5 | -1 | 5,8 | -5,4 |
| 1 | 7 | -14 | 2,8 | 2,9 | 1 | 10 | 6 | 1,4 | -2,2 | 2 | 2 | 6 | 11,1 | -10,7 | 2 | 5 | 0 | 3,2 | -2,5 |
| 1 | 7 | -13 | 4,8 | -5,3 | 1 | 10 | 7 | 2,0 | 1,5 | 2 | 2 | 7 | 6,4 | 7,1 | 2 | 5 | 1 | 3,5 | 3,9 |
| 1 | 7 | -12 | 6,1 | 7,1 | 1 | 10 | 8 | 1,4 | 1,3 | 2 | 2 | 8 | 4,8 | 5,0 | 2 | 5 | 2 | 8,6 | 11,0 |
| 1 | 7 | -11 | 5,6 | -6,3 | 1 | 10 | 9 | 2,4 | 3,2 | 2 | 2 | 9 | 14,7 | 14,3 | 2 | 5 | 3 | 6,0 | 7,0 |
| 1 | 7 | -10 | 2,9 | 3,1 | 1 | 10 | 10 | 1,9 | 2,1 | 2 | 2 | 10 | < 1,6 | 0,5 | 2 | 5 | 4 | 8,3 | 9,4 |
| 1 | 7 | -9 | 3,7 | 3,7 | 1 | 10 | 11 | 2,3 | 1,9 | 2 | 2 | 11 | 16,0 | 15,3 | 2 | 5 | 5 | 11,9 | 12,1 |
| 1 | 7 | -8 | 6,6 | -6,3 | 1 | 10 | 12 | < 1,2 | -0,3 | 2 | 2 | 12 | 8,2 | -8,1 | 2 | 5 | 6 | 5,0 | -6,0 |
| 1 | 7 | -7 | < 1,3 | 0,7 | 1 | 10 | 13 | 1,2 | 1,7 | 2 | 2 | 13 | 3,0 | 3,6 | 2 | 5 | 7 | 11,2 | 11,0 |
| 1 | 7 | -6 | 5,6 | -5,2 | 1 | 11 | -7 | 1,8 | 2,0 | 2 | 2 | 14 | 3,9 | -4,2 | 2 | 5 | 8 | 3,6 | -3,6 |
| 1 | 7 | -5 | 2,5 | 2,6 | 1 | 11 | -6 | 2,3 | 2,3 | 2 | 2 | 15 | 3,6 | -3,1 | 2 | 5 | 9 | 2,9 | 3,2 |
| 1 | 7 | -4 | 6,7 | -6,4 | 1 | 11 | -5 | 1,9 | -0,5 | 2 | 2 | 16 | < 1,7 | -0,6 | 2 | 5 | 10 | 1,7 | 1,3 |
| 1 | 7 | -3 | 8,8 | 8,1 | 1 | 11 | -4 | 1,4 | 1,6 | 2 | 2 | 17 | 3,0 | -2,7 | 2 | 5 | 11 | < 1,7 | -0,8 |
| 1 | 7 | -2 | 9,6 | -8,4 | 1 | 11 | -3 | < 1,4 | 0,1 | 2 | 2 | 18 | < 1,7 | -1,5 | 2 | 5 | 12 | < 1,7 | -0,1 |
| 1 | 7 | -1 | 19,2 | 18,4 | 1 | 11 | -2 | < 1,4 | -1,3 | 2 | 2 | 19 | < 1,7 | -1,3 | 2 | 5 | 13 | 3,0 | -2,9 |
| 1 | 7 | 0 | < 1,2 | 0,9 | 1 | 11 | -1 | 2,8 | 2,4 | 2 | 2 | 20 | 2,4 | -1,7 | 2 | 5 | 14 | 3,0 | -2,8 |
| 1 | 7 | 1 | 11,3 | 10,1 | 1 | 11 | 0 | < 1,4 | -0,6 | 2 | 3 | -15 | 4,6 | 3,7 | 2 | 6 | -16 | 3,9 | -3,9 |
| 1 | 7 | 2 | 8,1 | 8,2 | 1 | 11 | 1 | 1,4 | 1,6 | 2 | 3 | -14 | 8,0 | 7,8 | 2 | 6 | -15 | 3,9 | 3,8 |
| 1 | 7 | 3 | 4,7 | 5,1 | 1 | 11 | 2 | 2,0 | -2,1 | 2 | 3 | -13 | < 1,4 | 0,0 | 2 | 6 | -14 | < 1,6 | -0,9 |
| 1 | 7 | 4 | < 1,3 | -0,1 | 1 | 11 | 3 | < 1,4 | -0,6 | 2 | 3 | -12 | 4,5 | -4,5 | 2 | 6 | -13 | 4,5 | 3,9 |
| 1 | 7 | 5 | < 1,3 | 1,1 | 1 | 11 | 4 | 1,9 | -1,9 | 2 | 3 | -11 | 1,3 | 1,3 | 2 | 6 | -12 | 6,1 | 6,4 |
| 1 | 7 | 6 | < 1,3 | 1,0 | 1 | 11 | 5 | < 1,4 | 0,4 | 2 | 3 | -10 | 6,9 | -7,2 | 2 | 6 | -11 | 4,8 | -4,8 |
| 1 | 7 | 7 | 2,4 | 2,2 | 1 | 11 | 6 | < 1,3 | 0,4 | 2 | 3 | -9 | 8,9 | 8,8 | 2 | 6 | -10 | 3,0 | 3,3 |
| 1 | 7 | 8 | < 1,4 | 0,0 | 1 | 11 | 7 | < 1,3 | 1,9 | 2 | 3 | -8 | 16,3 | 15,9 | 2 | 6 | -9 | 12,0 | -10,9 |
| 1 | 7 | 9 | < 1,4 | 0,1 | 1 | 11 | 8 | 2,2 | 1,9 | 2 | 3 | -7 | 14,8 | 14,6 | 2 | 6 | -8 | < 1,5 | -0,9 |
| 1 | 7 | 10 | 4,1 | -4,9 | 1 | 11 | 9 | 1,7 | 1,6 | 2 | 3 | -6 | 22,3 | 22,5 | 2 | 6 | -7 | 11,3 | -10,8 |
| 1 | 7 | 11 | < 1,5 | -1,8 | 1 | 11 | 10 | 2,0 | 1,6 | 2 | 3 | -5 | 25,3 | -26,4 | 2 | 6 | -6 | 5,0 | 4,6 |
| 1 | 7 | 12 | 4,6 | -5,2 | 1 | 11 | 11 | < 1,1 | 0,6 | 2 | 3 | -4 | 1,3 | -0,3 | 2 | 6 | -5 | 7,7 | -8,0 |
| 1 | 7 | 13 | < 1,5 | 0,3 | 1 | 11 | 12 | 1,0 | 1,3 | 2 | 3 | -3 | 21,8 | -19,2 | 2 | 6 | -4 | 2,4 | 2,7 |
| 1 | 7 | 14 | 2,1 | 1,7 | 1 | 12 | -2 | 2,5 | -2,6 | 2 | 3 | -2 | 13,3 | -14,3 | 2 | 6 | -3 | 8,3 | -10,1 |
| 1 | 7 | 15 | 1,4 | 1,3 | 1 | 13 | -3 | 1,4 | -2,7 | 2 | 3 | -1 | 3,4 | -2,7 | 2 | 6 | -2 | 13,8 | -14,0 |
| 1 | 7 | 16 | 2,4 | 2,7 | 1 | 13 | -2 | < 1,0 | -0,2 | 2 | 3 | 0 | 2,3 | -4,2 | 2 | 6 | -1 | < 1,6 | -0,6 |
| 1 | 8 | -15 | 1,4 | -1,3 | 1 | 13 | -1 | 2,1 | -2,6 | 2 | 3 | 1 | 2,1 | -1,9 | 2 | 6 | 0 | 12,4 | -13,5 |
| 1 | 8 | -14 | 1,4 | -1,2 | 2 | 0 | -12 | 22,5 | -24,1 | 2 | 3 | 2 | 7,8 | -7,8 | 2 | 6 | 1 | 11,8 | 11,2 |
| 1 | 8 | -13 | < 1,4 | -0,5 | 2 | 0 | -10 | 7,5 | 8,4 | 2 | 3 | 3 | 12,4 | -13,3 | 2 | 6 | 2 | 2,6 | -2,9 |
| 1 | 8 | -12 | 11,1 | -10,4 | 2 | 0 | -8 | 8,0 | 9,7 | 2 | 3 | 4 | 4,1 | -4,4 | 2 | 6 | 3 | 7,2 | 8,4 |
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| 1 | 8 | -10 | 7,2 | -7,9 | 2 | 0 | -4 | 34,0 | 31,5 | 2 | 3 | 6 | 12,4 | 12,3 | 2 | 6 | 5 | < 1,6 | -0,6 |
| 1 | 8 | -9 | < 1,4 | 0,9 | 2 | 0 | -2 | --- | 66,9 | 2 | 3 | 7 | 4,3 | 5,1 | 2 | 6 | 6 | 4,5 | -6,0 |
| 1 | 8 | -8 | < 1,4 | 1,4 | 2 | 0 | 0 | 30,2 | 29,5 | 2 | 3 | 8 | 14,0 | 13,7 | 2 | 6 | 7 | 4,4 | -3,6 |
| 1 | 8 | -7 | 6,5 | -6,1 | 2 | 0 | 2 | 11,2 | -14,6 | 2 | 3 | 9 | < 1,6 | 1,2 | 2 | 6 | 8 | < 1,6 | -1,8 |
| 1 | 8 | -6 | < 1,4 | 0,5 | 2 | 0 | 4 | 15,0 | -13,1 | 2 | 3 | 10 | 9,9 | 9,8 | 2 | 6 | 9 | 2,4 | -2,5 |
| 1 | 8 | -5 | 6,3 | -6,3 | 2 | 0 | 6 | 17,4 | -18,3 | 2 | 3 | 11 | 4,7 | -5,2 | 2 | 6 | 10 | < 1,7 | -1,3 |
| 1 | 8 | -4 | < 1,3 | -1,1 | 2 | 0 | 8 | 8,4 | -8,1 | 2 | 3 | 12 | 6,4 | 6,9 | 2 | 6 | 11 | 3,8 | -3,4 |
| 1 | 8 | -3 | 3,5 | -3,5 | 2 | 0 | 10 | 4,4 | 5,8 | 2 | 3 | 13 | 4,1 | -3,5 | 2 | 6 | 12 | 1,7 | -1,8 |
| 1 | 8 | -2 | 18,0 | 15,0 | 2 | 0 | 12 | 5,9 | 6,3 | 2 | 3 | 14 | 1,7 | 1,4 | 2 | 6 | 13 | 2,4 | -1,7 |
| 1 | 8 | -1 | 1,3 | 1,7 | 2 | 0 | 14 | 3,5 | 3,2 | 2 | 3 | 15 | 1,7 | -2,6 | 2 | 7 | -15 | 2,1 | -2,3 |
| 1 | 8 | 0 | 18,5 | 15,9 | 2 | 0 | 16 | 5,0 | 4,4 | 2 | 3 | 16 | 3,5 | -3,2 | 2 | 7 | -14 | 5,5 | 6,2 |
| 1 | 8 | 1 | 2,7 | -2,8 | 2 | 0 | 18 | 2,4 | 2,3 | 2 | 3 | 17 | < 1,7 | -1,3 | 2 | 7 | -13 | 2,3 | -1,7 |
| 1 | 8 | 2 | 1,4 | -2,0 | 2 | 0 | 20 | 1,5 | -1,1 | 2 | 3 | 18 | 2,3 | -2,0 | 2 | 7 | -12 | 1,6 | 1,9 |
| 1 | 8 | 3 | < 1,4 | 0,6 | 2 | 1 | -14 | 14,1 | -14,2 | 2 | 4 | -18 | 4,5 | -4,4 | 2 | 7 | -11 | < 1,6 | 0,7 |
| 1 | 8 | 4 | 5,4 | -5,6 | 2 | 1 | -13 | 13,4 | -13,2 | 2 | 4 | -17 | < 1,6 | 1,7 | 2 | 7 | -10 | 7,4 | -6,8 |
| 1 | 8 | 5 | 2,4 | 2,2 | 2 | 1 | -12 | 10,4 | -11,3 | 2 | 4 | -16 | 5,0 | 3,8 | 2 | 7 | -9 | < 1,6 | 0,1 |
| 1 | 8 | 6 | 4,3 | -4,6 | 2 | 1 | -11 | 17,0 | -16,6 | 2 | 4 | -15 | < 1,7 | 0,5 | 2 | 7 | -8 | 4,5 | -4,6 |
| 1 | 8 | 7 | 2,5 | 2,6 | 2 | 1 | -10 | 8,1 | 8,2 | 2 | 4 | -14 | 9,3 | 8,7 | 2 | 7 | -7 | 2,2 | -3,3 |

| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
| 2 | 7 | -6 | 3,8 | 4,1 |
| 2 | 7 | -5 | 4,1 | 3,4 |
| 2 | 7 | -4 | 3,4 | -3,8 |
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| 2 | 8 | -8 | 1,6 | -1,7 |
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| 2 | 8 | -1 | 2,3 | 2,1 |
| 2 | 8 | 0 | < 1,6 | -0,7 |
| 2 | 8 | 1 | < 1,6 | 1,7 |
| 2 | 8 | 2 | 4,1 | -3,8 |
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| 2 | 8 | 8 | 3,4 | -3,9 |
| 2 | 8 | 9 | 2,4 | -2,0 |
| 2 | 8 | 10 | < 1,6 | 0,5 |
| 2 | 8 | 11 | < 1,6 | 0,8 |
| 2 | 8 | 12 | 3,7 | 3,3 |
| 2 | 8 | 13 | < 1,5 | 0,6 |
| 2 | 8 | 14 | 2,1 | 2,1 |
| 2 | 9 | -10 | 5,3 | 5,0 |
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| 2 | 9 | -8 | 2,3 | 3,4 |
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| 2 | 10 | -9 | 3,1 | 2,5 |
| 2 | 10 | -8 | 2,2 | -2,6 |
| 2 | 10 | -7 | 3,5 | 3,7 |
| 2 | 10 | -6 | < 1,6 | -0,1 |
| 2 | 10 | -5 | < 1,6 | 1,3 |
| 2 | 10 | -4 | 2,3 | 2,3 |
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| 2 | 10 | -2 | < 1,6 | 0,2 |
| 2 | 10 | -1 | < 1,6 | -0,4 |
| 2 | 10 | 0 | < 1,6 | -0,7 |
| 2 | 10 | 1 | < 1,6 | -2,9 |
| 2 | 10 | 2 | < 1,6 | -0,6 |
| 2 | 10 | 3 | 3,3 | -3,8 |
| 2 | 10 | 4 | 1,6 | -1,8 |
| 2 | 10 | 5 | < 1,6 | -1,7 |
| 2 | 10 | 6 | 1,6 | -1,4 |
| 2 | 11 | -9 | 1,2 | -2,0 |
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| 2 | 11 | -5 | 2,6 | -2,5 |
| 2 | 11 | -4 | 1,5 | 1,6 |
| 2 | 11 | -3 | < 1,5 | -1,4 |
| 2 | 11 | -2 | < 1,5 | 0,6 |
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| 3 | 0 | -12 | 3,6 | -4,0 |
| 3 | 0 | -10 | 2,3 | -2,7 |
| 3 | 0 | -8 | 8,3 | -8,1 |
| 3 | 0 | -6 | 6,0 | 6,5 |
| 3 | 0 | -4 | 15,0 | 16,3 |
| 3 | 0 | -2 | —— | 8,5 |
| 3 | 0 | 0 | 5,5 | -5,9 |
| 3 | 0 | 2 | 19,3 | -18,2 |
| 3 | 0 | 4 | 13,0 | -13,6 |
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| 3 | 0 | 8 | 6,9 | -6,1 |
| 3 | 0 | 10 | < 2,3 | -0,7 |
| 3 | 0 | 12 | 5,9 | 4,6 |
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| 3 | 1 | -14 | 4,5 | -5,5 |
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| 3 | 1 | -8 | < 1,5 | -0,6 |
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| 3 | 1 | -5 | 17,1 | 18,0 |
| 3 | 1 | -4 | 9,6 | 10,3 |
| 3 | 1 | -3 | 9,3 | 8,7 |
| 3 | 1 | -2 | 4,2 | -4,0 |
| 3 | 1 | -1 | 5,6 | -6,9 |
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| 3 | 1 | 1 | 12,7 | -13,4 |
| 3 | 1 | 2 | 1,5 | 1,9 |
| 3 | 1 | 3 | 12,5 | -12,0 |
| 3 | 1 | 4 | 7,7 | 8,3 |
| 3 | 1 | 5 | 8,7 | -8,0 |
| 3 | 1 | 6 | 3,7 | 5,0 |
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| 3 | 1 | 8 | 7,6 | 8,1 |
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| 3 | 1 | 10 | 6,9 | 6,6 |
| 3 | 1 | 11 | 4,7 | 4,8 |
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| 3 | 1 | 13 | 6,4 | 5,6 |
| 3 | 1 | 14 | 2,3 | -1,4 |
| 3 | 1 | 15 | 3,1 | 2,2 |
| 3 | 2 | -16 | 2,1 | -2,5 |
| 3 | 2 | -15 | 6,7 | -6,7 |
| 3 | 2 | -14 | 5,4 | 5,8 |
| 3 | 2 | -13 | 3,4 | -4,1 |
| 3 | 2 | -12 | 12,1 | 12,3 |
| 3 | 2 | -11 | 14,1 | 12,9 |
| 3 | 2 | -10 | < 1,7 | -1,8 |
| 3 | 2 | -9 | 15,7 | 16,5 |
| 3 | 2 | -8 | < 1,6 | -1,4 |
| 3 | 2 | -7 | 12,1 | 12,5 |
| 3 | 2 | -6 | 10,7 | 10,9 |
| 3 | 2 | -5 | 12,5 | 12,2 |
| 3 | 2 | -4 | 8,7 | 8,3 |
| 3 | 2 | -3 | 4,6 | 4,1 |
| 3 | 2 | -2 | 3,4 | -4,0 |
| 3 | 2 | -1 | 7,6 | -8,9 |
| 3 | 2 | 0 | 2,6 | -2,7 |
| 3 | 2 | 1 | 9,4 | -10,0 |
| 3 | 2 | 2 | < 1,6 | 0,7 |
| 3 | 2 | 3 | 4,5 | -5,5 |
| 3 | 2 | 4 | 2,6 | -2,7 |
| 3 | 2 | 5 | 2,7 | -3,0 |
| 3 | 2 | 6 | 2,0 | 1,2 |
| 3 | 2 | 7 | 4,1 | 4,3 |
| 3 | 2 | 8 | 3,0 | 3,8 |
| 3 | 2 | 9 | 6,5 | 6,7 |
| 3 | 2 | 10 | 2,3 | 1,4 |
| 3 | 2 | 11 | < 2,3 | 0,8 |
| 3 | 2 | 12 | 3,2 | 2,4 |
| 3 | 2 | 13 | 2,3 | -2,5 |
| 3 | 2 | 14 | 4,5 | 3,7 |
| 3 | 3 | -18 | 4,1 | -3,7 |
| 3 | 3 | -17 | 2,0 | 1,8 |
| 3 | 3 | -16 | 5,8 | -6,0 |
| 3 | 3 | -15 | < 2,1 | 2,7 |
| 3 | 3 | -14 | 5,0 | -5,7 |
| 3 | 3 | -13 | < 2,0 | 0,9 |
| 3 | 3 | -12 | 2,8 | -3,5 |
| 3 | 3 | -11 | 3,8 | 4,3 |
| 3 | 3 | -10 | 6,7 | 7,0 |
| 3 | 3 | -9 | < 1,8 | 1,0 |
| 3 | 3 | -8 | 11,8 | 11,3 |
| 3 | 3 | -7 | 7,9 | -7,8 |
| 3 | 3 | -6 | 2,7 | 2,7 |
| 3 | 3 | -5 | 11,7 | -10,8 |
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| 3 | 3 | -3 | 5,1 | 5,5 |
| 3 | 3 | -2 | < 1,6 | -1,4 |
| 3 | 3 | -1 | 6,0 | 4,1 |
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| 3 | 3 | 1 | < 1,7 | 1,6 |
| 3 | 3 | 2 | 18,5 | -17,2 |
| 3 | 3 | 3 | 10,2 | 11,4 |
| 3 | 3 | 4 | 8,5 | -8,7 |
| 3 | 3 | 5 | 12,0 | 12,1 |
| 3 | 3 | 6 | 4,0 | 4,3 |
| 3 | 3 | 7 | 3,0 | 2,4 |
| 3 | 3 | 8 | 3,1 | 2,7 |
| 3 | 3 | 9 | 2,3 | -2,4 |
| 3 | 3 | 10 | 2,3 | -2,9 |
| 3 | 3 | 11 | 2,3 | -2,0 |
| 3 | 4 | -18 | 2,8 | -2,7 |
| 3 | 4 | -17 | < 2,1 | -1,0 |
| 3 | 4 | -16 | 5,1 | 4,3 |
| 3 | 4 | -15 | 5,5 | -5,3 |
| 3 | 4 | -14 | < 2,1 | 1,7 |
| 3 | 4 | -13 | 2,9 | -3,2 |
| 3 | 4 | -12 | < 2,1 | 0,1 |
| 3 | 4 | -11 | 2,0 | 1,4 |
| 3 | 4 | -10 | 4,8 | 5,7 |
| 3 | 4 | -9 | 1,9 | 2,0 |
| 3 | 4 | -8 | 2,7 | -1,4 |
| 3 | 4 | -7 | 3,2 | -3,1 |
| 3 | 4 | -6 | 16,3 | -15,2 |
| 3 | 4 | -5 | 1,8 | -1,8 |
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| 3 | 4 | -3 | 1,7 | 1,2 |
| 3 | 4 | -2 | < 1,7 | -2,1 |
| 3 | 4 | -1 | 1,7 | -2,1 |
| 3 | 4 | 0 | 5,4 | -6,3 |
| 3 | 4 | 1 | 3,7 | -4,0 |
| 3 | 4 | 2 | 1,9 | 2,1 |
| 3 | 4 | 3 | 3,9 | -4,3 |
| 3 | 4 | 4 | 13,3 | 14,4 |
| 3 | 4 | 5 | 2,1 | -1,7 |
| 3 | 4 | 6 | 5,6 | 6,1 |
| 3 | 4 | 7 | 2,1 | 1,6 |
| 3 | 4 | 8 | 5,0 | -4,7 |
| 3 | 4 | 9 | < 2,3 | -0,7 |
| 3 | 4 | 10 | 3,2 | -3,4 |
| 3 | 4 | 11 | 2,3 | -2,8 |
| 3 | 4 | 12 | 2,3 | -2,1 |
| 3 | 4 | 13 | 2,2 | -1,9 |
| 3 | 4 | 14 | 4,1 | -3,6 |
| 3 | 4 | 15 | < 2,0 | -0,5 |
| 3 | 4 | 16 | 1,9 | -1,8 |
| 3 | 5 | -19 | 3,2 | -2,5 |
| 3 | 5 | -18 | 3,4 | 2,2 |
| 3 | 5 | -17 | < 2,0 | -1,5 |
| 3 | 5 | -16 | < 2,1 | 0,7 |
| 3 | 5 | -15 | 2,1 | 1,8 |
| 3 | 5 | -14 | 3,7 | -2,8 |
| 3 | 5 | -13 | 2,1 | 1,9 |
| 3 | 5 | -12 | < 2,1 | 0,4 |
| 3 | 5 | -11 | < 2,1 | 2,3 |
| 3 | 5 | -10 | 2,1 | 1,7 |
| 3 | 5 | -9 | 4,0 | 4,4 |
| 3 | 5 | -8 | < 2,0 | 1,1 |
| 3 | 5 | -7 | 3,9 | -2,8 |
| 3 | 5 | -6 | < 1,9 | 0,7 |
| 3 | 5 | -5 | 8,6 | -9,2 |
| 3 | 5 | -4 | 9,1 | 8,5 |
| 3 | 5 | -3 | 5,7 | -7,5 |
| 3 | 5 | -2 | 11,2 | 12,4 |
| 3 | 5 | -1 | 3,9 | -3,3 |
| 3 | 5 | 0 | 8,2 | 8,4 |
| 3 | 5 | 1 | < 2,0 | 0,3 |
| 3 | 5 | 2 | 2,0 | 2,1 |
| 3 | 5 | 3 | 4,6 | 4,3 |
| 3 | 5 | 4 | < 2,1 | -1,0 |
| 3 | 5 | 5 | 6,0 | 5,1 |
| 3 | 5 | 6 | 3,1 | -2,8 |
| 3 | 5 | 7 | 2,3 | 2,0 |
| 3 | 5 | 8 | 2,3 | -3,2 |
| 3 | 5 | 9 | < 2,3 | -1,0 |
| 3 | 5 | 10 | 3,2 | -3,1 |
| 3 | 5 | 11 | 3,3 | -2,6 |
| 3 | 5 | 12 | 3,1 | -3,2 |
| 3 | 5 | 13 | 3,7 | -3,2 |
| 3 | 6 | -16 | 3,6 | -2,8 |
| 3 | 6 | -15 | < 2,1 | 1,8 |
| 3 | 6 | -14 | < 2,1 | -0,4 |
| 3 | 6 | -13 | < 2,1 | 0,7 |
| 3 | 6 | -12 | < 2,1 | 0,2 |
| 3 | 6 | -11 | < 2,1 | -0,8 |
| 3 | 6 | -10 | 3,0 | -3,0 |
| 3 | 6 | -9 | < 2,1 | 0,1 |
| 3 | 6 | -8 | < 2,1 | 0,3 |
| 3 | 6 | -7 | < 2,1 | -1,1 |
| 3 | 6 | -6 | 5,1 | 4,7 |
| 3 | 6 | -5 | < 2,1 | 0,5 |
| 3 | 6 | -4 | < 2,1 | 1,1 |
| 3 | 6 | -3 | 9,8 | 8,6 |
| 3 | 6 | -2 | < 2,1 | -1,1 |
| 3 | 6 | -1 | 16,0 | 14,8 |
| 3 | 6 | 0 | 6,8 | 6,7 |
| 3 | 6 | 1 | 7,8 | 6,9 |
| 3 | 6 | 2 | 8,7 | 9,4 |
| 3 | 6 | 3 | < 2,1 | -1,6 |
| 3 | 6 | 4 | 3,8 | 3,5 |
| 3 | 6 | 5 | 2,3 | -1,0 |
| 3 | 6 | 6 | 2,3 | 3,0 |
| 3 | 6 | 7 | 2,3 | -1,5 |
| 3 | 6 | 8 | 2,3 | 2,3 |
| 3 | 6 | 9 | 3,9 | -4,4 |
| 3 | 6 | 10 | < 2,3 | -0,7 |
| 3 | 6 | 11 | 3,8 | -3,4 |
| 3 | 6 | 12 | 3,8 | -2,0 |
| 3 | 7 | -15 | 2,0 | -2,7 |
| 3 | 7 | -14 | 2,9 | 2,9 |
| 3 | 7 | -13 | 4,2 | -3,0 |
| 3 | 7 | -12 | 3,0 | -2,0 |
| 3 | 7 | -11 | < 2,1 | -1,9 |
| 3 | 7 | -10 | 4,3 | -4,5 |
| 3 | 7 | -9 | 2,1 | 1,0 |
| 3 | 7 | -8 | 3,1 | -3,3 |
| 3 | 7 | -7 | 4,3 | 4,0 |
| 3 | 7 | -6 | 6,8 | -6,2 |
| 3 | 7 | -5 | 5,7 | 5,1 |
| 3 | 7 | -4 | 9,0 | -8,2 |
| 3 | 7 | -3 | 4,3 | 4,0 |
| 3 | 7 | -2 | 3,8 | 2,7 |
| 3 | 7 | -1 | 6,3 | 5,6 |
| 3 | 7 | 0 | 7,6 | 7,0 |
| 3 | 7 | 1 | 3,1 | 4,0 |
| 3 | 7 | 2 | < 2,3 | -0,1 |
| 3 | 7 | 3 | < 2,3 | 1,0 |
| 3 | 7 | 4 | < 2,3 | -1,2 |
| 3 | 7 | 5 | < 2,3 | 0,1 |
| 3 | 7 | 6 | < 2,3 | 0,9 |
| 3 | 7 | 7 | < 2,3 | 0,9 |
| 3 | 7 | 8 | < 2,2 | -1,9 |
| 3 | 7 | 9 | < 2,1 | 1,0 |
| 3 | 7 | 10 | 3,7 | -2,7 |
| 3 | 8 | -15 | 3,1 | -1,2 |
| 3 | 8 | -14 | 1,9 | -2,6 |
| 3 | 8 | -13 | < 2,0 | 0,1 |
| 3 | 8 | -12 | 4,6 | -4,0 |
| 3 | 8 | -11 | < 2,1 | 0,5 |
| 3 | 8 | -10 | < 2,1 | -0,6 |
| 3 | 8 | -9 | 4,3 | -3,9 |
| 3 | 8 | -8 | 3,1 | 2,7 |
| 3 | 8 | -7 | 5,7 | -6,3 |
| 3 | 8 | -6 | 3,1 | 3,1 |
| 3 | 8 | -5 | 4,9 | -4,4 |
| 3 | 8 | -4 | 2,2 | 2,2 |
| 3 | 8 | -3 | 2,2 | -1,6 |
| 3 | 8 | -2 | 2,2 | 2,5 |
| 3 | 8 | -1 | 2,2 | -1,8 |
| 3 | 8 | 0 | < 2,2 | 0,7 |
| 3 | 8 | 1 | 2,2 | -1,6 |
| 3 | 8 | 2 | 3,1 | -2,3 |
| 3 | 8 | 3 | < 2,2 | -0,4 |
| 3 | 8 | 4 | 4,5 | -4,0 |
| 3 | 8 | 5 | < 2,3 | -0,7 |
| 3 | 8 | 6 | 3,1 | -2,8 |
| 3 | 9 | -11 | 4,5 | -3,7 |
| 3 | 9 | -10 | 2,0 | 2,3 |
| 3 | 9 | -9 | 2,9 | -2,7 |
| 3 | 9 | -8 | 2,1 | -0,8 |
| 3 | 9 | -7 | < 2,1 | 1,6 |
| 3 | 9 | -6 | < 2,1 | -1,5 |
| 3 | 9 | -5 | 2,1 | 2,2 |
| 3 | 9 | -4 | < 2,1 | -0,3 |
| 3 | 9 | -3 | < 2,1 | 0,9 |
| 3 | 9 | -2 | < 2,1 | -1,8 |
| 3 | 9 | -1 | < 2,1 | 1,4 |
| 3 | 9 | 0 | 2,1 | -2,6 |
| 3 | 9 | 1 | 2,1 | 1,7 |
| 3 | 9 | 2 | < 2,1 | -0,4 |
| 3 | 9 | 3 | 3,0 | -2,0 |
| 3 | 9 | 4 | < 2,1 | 0,2 |
| 3 | 9 | 5 | 3,6 | -3,4 |
| 3 | 10 | -4 | 2,0 | 0,8 |
| 3 | 10 | -3 | < 2,0 | -1,8 |
| 3 | 10 | -2 | 2,0 | 1,3 |
| 3 | 10 | -1 | < 2,0 | -2,4 |
| 3 | 10 | 0 | 2,8 | 2,8 |
| 3 | 10 | 1 | 2,6 | -0,9 |
| 3 | 10 | 2 | 1,9 | 1,2 |
| 3 | 10 | 3 | < 1,9 | 1,0 |
| 3 | 10 | 4 | 1,9 | -1,3 |
| 3 | 10 | 5 | 2,5 | 1,9 |
| 3 | 10 | 6 | < 1,8 | -0,7 |
| 3 | 10 | 7 | 3,2 | 2,5 |
| 4 | 0 | -8 | 8,3 | 8,1 |
| 4 | 0 | -6 | 3,2 | 2,8 |
| 4 | 0 | -4 | 1,1 | 0,8 |
| 4 | 0 | -2 | 6,2 | 5,2 |
| 4 | 0 | 0 | 3,6 | 2,8 |
| 4 | 0 | 2 | 6,7 | -5,3 |
| 4 | 0 | 4 | 3,6 | -2,8 |
| 4 | 0 | 6 | 3,0 | 3,6 |
| 4 | 0 | 8 | 4,5 | 4,7 |
| 4 | 0 | 10 | 4,7 | 4,7 |
| 4 | 0 | 12 | 2,0 | 2,7 |
| 4 | 1 | -13 | 8,2 | -8,2 |
| 4 | 1 | -12 | 5,4 | 4,8 |
| 4 | 1 | -11 | 7,4 | -8,7 |
| 4 | 1 | -10 | < 1,7 | -0,4 |
| 4 | 1 | -9 | < 1,6 | 0,3 |
| 4 | 1 | -8 | 4,8 | -5,0 |
| 4 | 1 | -7 | 8,1 | 7,8 |
| 4 | 1 | -6 | 2,1 | 3,1 |
| 4 | 1 | -5 | 5,5 | 6,2 |
| 4 | 1 | -4 | 5,1 | -5,1 |

| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
| 4 | 1 | -3 | < 1.4 | 1.3 |
| 4 | 1 | -2 | 14.3 | -14.8 |
| 4 | 1 | -1 | 5.3 | 4.3 |
| 4 | 1 | 0 | 11.4 | -10.8 |
| 4 | 1 | 1 | 4.1 | 3.3 |
| 4 | 1 | 2 | 2.6 | -3.0 |
| 4 | 1 | 3 | 3.4 | -2.3 |
| 4 | 1 | 4 | < 2.0 | -1.1 |
| 4 | 1 | 5 | 4.1 | -3.4 |
| 4 | 1 | 6 | < 2.1 | -0.1 |
| 4 | 1 | 7 | < 2.1 | 0.7 |
| 4 | 1 | 8 | 3.1 | 2.0 |
| 4 | 1 | 9 | 3.8 | 3.3 |
| 4 | 1 | 10 | < 2.1 | 1.4 |
| 4 | 1 | 11 | 2.1 | 1.9 |
| 4 | 2 | -13 | 3.7 | 3.1 |
| 4 | 2 | -12 | 4.1 | -4.8 |
| 4 | 2 | -11 | < 1.8 | 0.2 |
| 4 | 2 | -10 | 6.4 | -6.7 |
| 4 | 2 | -9 | 3.9 | -3.4 |
| 4 | 2 | -8 | 2.4 | 2.0 |
| 4 | 2 | -7 | < 1.6 | 1.0 |
| 4 | 2 | -6 | 2.3 | 3.0 |
| 4 | 2 | -5 | 5.3 | -4.0 |
| 4 | 2 | -4 | 3.1 | -3.4 |
| 4 | 2 | -3 | 16.5 | -15.2 |
| 4 | 2 | -2 | < 1.6 | 1.4 |
| 4 | 2 | -1 | 9.7 | -10.3 |
| 4 | 2 | 0 | 6.7 | 6.6 |
| 4 | 2 | 1 | < 1.8 | -0.9 |
| 4 | 2 | 2 | 1.9 | 1.7 |
| 4 | 2 | 3 | < 1.8 | 2.4 |
| 4 | 2 | 4 | 4.1 | -3.5 |
| 4 | 2 | 5 | 3.0 | 3.7 |
| 4 | 2 | 6 | < 2.1 | -1.3 |
| 4 | 2 | 7 | 6.5 | 5.9 |
| 4 | 2 | 8 | < 2.2 | 0.7 |
| 4 | 2 | 9 | 5.3 | 4.1 |
| 4 | 2 | 10 | < 2.1 | -0.9 |
| 4 | 2 | 11 | < 2.1 | 0.6 |
| 4 | 2 | 12 | 2.0 | -1.7 |
| 4 | 3 | -14 | 5.0 | 4.8 |
| 4 | 3 | -13 | < 1.9 | 0.2 |
| 4 | 3 | -12 | 2.7 | 2.3 |
| 4 | 3 | -11 | 1.8 | 2.1 |
| 4 | 3 | -10 | < 1.8 | 0.7 |
| 4 | 3 | -9 | 1.8 | -2.1 |
| 4 | 3 | -8 | 2.6 | 3.3 |
| 4 | 3 | -7 | 5.4 | -5.1 |
| 4 | 3 | -6 | < 1.7 | 1.9 |
| 4 | 3 | -5 | 6.5 | -5.8 |
| 4 | 3 | -4 | 3.5 | -3.2 |
| 4 | 3 | -3 | 3.5 | -3.4 |
| 4 | 3 | -2 | 6.7 | -5.8 |
| 4 | 3 | -1 | < 1.8 | -1.0 |
| 4 | 3 | 0 | 3.8 | -3.9 |
| 4 | 3 | 1 | 4.1 | 4.0 |
| 4 | 3 | 2 | 2.0 | 1.7 |
| 4 | 3 | 3 | 5.0 | 4.3 |
| 4 | 3 | 4 | 5.5 | 6.8 |
| 4 | 3 | 5 | < 2.1 | 1.0 |
| 4 | 3 | 6 | 7.1 | 6.7 |
| 4 | 3 | 7 | < 2.1 | 0.5 |
| 4 | 3 | 8 | 4.9 | 3.9 |
| 4 | 3 | 9 | < 2.1 | 0.3 |
| 4 | 3 | 10 | 3.0 | 2.6 |
| 4 | 3 | 11 | 2.0 | -1.3 |
| 4 | 3 | 12 | 1.9 | 1.8 |
| 4 | 4 | -16 | 3.5 | 3.1 |
| 4 | 4 | -15 | 1.8 | 1.8 |
| 4 | 4 | -14 | < 1.8 | -0.3 |
| 4 | 4 | -13 | 2.7 | 3.0 |
| 4 | 4 | -12 | 1.9 | 2.5 |
| 4 | 4 | -11 | < 1.9 | 0.0 |
| 4 | 4 | -10 | 3.4 | 3.6 |
| 4 | 4 | -9 | < 1.9 | 0.3 |
| 4 | 4 | -8 | 5.0 | -3.8 |
| 4 | 4 | -7 | 3.8 | 3.7 |
| 4 | 4 | -6 | 6.3 | -5.9 |
| 4 | 4 | -5 | 3.3 | 2.9 |
| 4 | 4 | -4 | < 1.9 | -0.9 |
| 4 | 4 | -3 | < 1.9 | -2.4 |
| 4 | 4 | -2 | < 1.9 | -0.5 |
| 4 | 4 | -1 | 4.8 | -4.8 |
| 4 | 4 | 0 | < 2.0 | 1.2 |
| 4 | 4 | 1 | < 2.0 | -0.4 |
| 4 | 4 | 2 | 8.2 | 7.1 |
| 4 | 4 | 3 | < 2.1 | 1.7 |
| 4 | 4 | 4 | 5.6 | 5.8 |
| 4 | 4 | 5 | < 2.1 | 1.0 |
| 4 | 4 | 6 | < 2.1 | 0.3 |
| 4 | 4 | 7 | < 2.1 | 1.0 |
| 4 | 4 | 8 | < 2.1 | -1.4 |
| 4 | 4 | 9 | 3.0 | 2.2 |
| 4 | 4 | 10 | < 2.0 | -0.4 |
| 4 | 4 | 11 | 2.8 | 2.3 |
| 4 | 5 | -12 | 3.5 | -3.8 |
| 4 | 5 | -11 | 2.0 | 3.3 |
| 4 | 5 | -10 | 5.9 | -5.6 |
| 4 | 5 | -9 | < 2.0 | 1.5 |
| 4 | 5 | -8 | 5.6 | -5.9 |
| 4 | 5 | -7 | 3.4 | -2.6 |
| 4 | 5 | -6 | < 2.0 | -2.0 |
| 4 | 5 | -5 | 2.0 | -2.0 |
| 4 | 5 | -4 | < 2.0 | 0.9 |
| 4 | 5 | -3 | < 2.0 | -0.8 |
| 4 | 5 | -2 | 2.9 | -2.9 |
| 4 | 5 | -1 | 2.0 | 0.6 |
| 4 | 5 | 0 | 2.1 | -2.1 |
| 4 | 5 | 1 | 3.0 | 2.9 |
| 4 | 5 | 2 | 2.1 | 2.1 |
| 4 | 5 | 3 | 3.0 | 2.4 |
| 4 | 5 | 4 | < 2.1 | 0.4 |
| 4 | 5 | 5 | < 2.1 | 0.5 |
| 4 | 5 | 6 | 2.7 | -3.5 |
| 4 | 5 | 7 | < 2.1 | -0.5 |
| 4 | 5 | 8 | 2.1 | -1.7 |
| 4 | 6 | -15 | 1.7 | 1.3 |
| 4 | 6 | -14 | 3.1 | 3.0 |
| 4 | 6 | -13 | 3.2 | -2.9 |
| 4 | 6 | -12 | 2.7 | 3.5 |
| 4 | 6 | -11 | 5.4 | -6.7 |
| 4 | 6 | -10 | < 1.9 | 1.6 |
| 4 | 6 | -9 | 5.1 | -5.0 |
| 4 | 6 | -8 | < 1.9 | 1.1 |
| 4 | 6 | -7 | 4.1 | -4.1 |
| 4 | 6 | -6 | < 2.0 | -0.3 |
| 4 | 6 | -5 | < 2.0 | -1.5 |
| 4 | 6 | -4 | 4.1 | -4.0 |
| 4 | 6 | -3 | 2.1 | 1.4 |
| 4 | 6 | -2 | 4.6 | -4.8 |
| 4 | 6 | -1 | 2.1 | 1.6 |
| 4 | 6 | 0 | 3.0 | -2.5 |
| 4 | 6 | 1 | < 2.1 | 0.4 |
| 4 | 6 | 2 | 2.1 | -2.2 |
| 4 | 6 | 3 | < 2.1 | 0.0 |
| 4 | 6 | 4 | 2.1 | -1.4 |
| 4 | 6 | 5 | 2.1 | -1.3 |
| 4 | 6 | 6 | < 2.1 | 0.0 |
| 4 | 6 | 7 | 4.3 | -3.5 |
| 4 | 6 | 8 | < 2.0 | -0.2 |
| 4 | 6 | 9 | 1.9 | -2.2 |
| 4 | 7 | -13 | 1.7 | 1.5 |
| 4 | 7 | -12 | 2.6 | -1.6 |
| 4 | 7 | -11 | 1.8 | 2.4 |
| 4 | 7 | -10 | < 1.8 | -0.9 |
| 4 | 7 | -9 | 1.9 | 2.3 |
| 4 | 7 | -8 | 2.0 | 1.2 |
| 4 | 7 | -7 | 2.0 | 1.0 |
| 4 | 7 | -6 | < 2.0 | 1.0 |
| 4 | 7 | -5 | < 2.0 | 0.4 |
| 4 | 7 | -4 | 2.0 | 1.7 |
| 4 | 7 | -3 | < 2.1 | -0.5 |
| 4 | 7 | -2 | 3.6 | 3.4 |
| 4 | 7 | -1 | 2.9 | -3.1 |
| 4 | 7 | 0 | 2.1 | 2.1 |
| 4 | 7 | 1 | 5.1 | -5.2 |
| 4 | 7 | 2 | < 2.1 | 0.9 |
| 4 | 7 | 3 | 2.9 | -2.8 |
| 4 | 7 | 4 | < 2.0 | -0.2 |
| 4 | 7 | 5 | < 2.0 | -0.8 |
| 4 | 7 | 6 | < 1.9 | -2.3 |
| 4 | 7 | 7 | < 1.9 | -0.8 |
| 4 | 7 | 8 | 1.7 | -2.1 |
| 4 | 7 | 9 | 1.7 | -0.2 |
| 4 | 7 | 10 | 1.6 | 0.2 |
| 4 | 8 | -8 | 2.4 | 2.4 |
| 4 | 8 | -7 | 1.9 | 1.5 |
| 4 | 8 | -6 | 1.9 | 2.4 |
| 4 | 8 | -5 | 1.9 | 2.3 |
| 4 | 8 | -4 | 2.8 | 2.7 |
| 4 | 8 | -3 | 1.9 | 1.6 |
| 4 | 8 | -2 | 2.0 | 1.7 |
| 4 | 8 | -1 | < 2.0 | 1.9 |
| 4 | 8 | 0 | 2.0 | -2.0 |
| 4 | 8 | 1 | 2.0 | 2.5 |
| 4 | 8 | 2 | 3.4 | -3.6 |
| 4 | 8 | 3 | 1.9 | 1.7 |
| 4 | 8 | 4 | 1.9 | -1.8 |
| 5 | 0 | -10 | 11.7 | 7.9 |
| 5 | 0 | -8 | 6.0 | 2.8 |
| 5 | 0 | -6 | < 2.3 | 1.2 |
| 5 | 0 | -4 | 6.1 | 5.5 |
| 5 | 0 | -2 | < 2.3 | -0.3 |
| 5 | 0 | 0 | 5.9 | -9.0 |
| 5 | 0 | 2 | 5.5 | -7.8 |
| 5 | 1 | -2 | 2.9 | 2.1 |
| 5 | 1 | -1 | 5.3 | -4.0 |
| 5 | 1 | 0 | 5.4 | 3.4 |
| 5 | 1 | 1 | 4.4 | -6.2 |

# THE CRYSTAL STRUCTURE OF THE METHYLZINC T-BUTYLSULPHIDE PENTAMER

## Introduction

The reaction between equimolar quantities of a dialkylzinc (Coates and Ridley, 1965) or dialkylcadmium (Coates and Lauder, 1966) and a compound containing active hydrogen yields a hydrocarbon and a derivative of the metal alkyl. Thus the reaction between dimethylyzinc and t-butylthiol gives methane and methylzinc t-butylsulphide.

$$Me_2Zn + Bu^tSH \rightarrow MeZnSBu^t + CH_4$$

The reactions of $Me_2Zn$ and $Me_2Cd$ with alcohols produce alkoxides, which are all tetrameric except methylcadmium t-butoxide which is dimeric. The molecular weights were determined cryoscopically in benzene. An x-ray structure analysis (Shearer and Spencer, 1966.a) has shown that $(MeZnOMe)_4$ is also tetrameric in the crystalline state, with zinc and oxygen atoms near the corners of a distorted cube. The molecular symmetry approximates to $\bar{4}3m$. The X-ray powder diffraction patterns of $(MeZnOMe)_4$ and $(MeCdOMe)_4$ are similar (Shearer and Spencer, 1966b) and crystals of the compounds are probably isomorphous. It is possible that all of the tetrameric alkoxides of dimethylzinc and dimethylcadmium are isostructural with $(MeZnOMe)_4$. The reaction of acetoxime with dimethylzinc (Coates and Ridley, 1966) gives a compound which is also tetrameric in benzene solution.

Dimthylzinc reacts with diphenylamine to give the dimeric compound $(MeZnNPh_2)_2$. In the crystal the molecule contains a four-membered zinc-nitrogen ring in which the zinc atoms are three-coordinate (Shearer and Spencer, 1966.c.). It seems likely that $(MeCdOBu^t)_2$ will have a similar structure.

The products of reactions with thiols show differing degrees of association. Methyl methylzinc sulphide is insoluble in benzene and is assumed to be polymeric in the solid state. Methylzinc i-propylsulphide and methylcadmium i-propylsulphide were found to be hexameric in benzene solution but methylzinc t-butylsulphide and ethylzinc t-butylsulphide are pentameric and methylcadmium t-butylsulphide is tetrameric. All of the molecular weight measurements were made at more than one concentration.

In a preliminary x-ray study of $(MeZnSBu^t)_5$ (Shearer and Willis, 1965) the size and contents of the unit cell were determined but the space group was not unambiguously identified. These observations were consistent with there being five or ten units of $MeZnSBu^t$ in the assymmetric unit. The crystal structure of this compound was determined with the object of finding the atomic arrangement and the reasons for its degree of association. It was thought that this information would help in explaining the different degrees of association and structures found in the other compounds mentioned above.

## Experimental

### Preparation of Crystals and Preliminary X-ray Examination

Methylzinc t-butylsulphide was prepared by adding a solution of t-butylthiol to an equimolar quantity of dimethylzinc in hexane. Crystals suitable for the structure analysis were obtained by recrystallisation from hexane and were sealed individually in pyrex glass capillary tubes in the dry nitrogen atmosphere of a glove-box. The crystals were in the form of transparent needles, elongated along $\underline{a}$ and with an eight-sided cross-section.

The unit cell dimensions were determined from precession photographs of the h0l, h1l, and hk0 reciprocal lattice nets taken with Mo. K$\alpha$ radiation. The standard deviations of the unit cell lengths are of $\underline{a}$, 0.004, of $\underline{b}$ 0.06 and of $\underline{c}$, 0.005Å. However if systematic errors are included, the uncertainty in these lengths is probably of the order of 0.25%. The standard deviation of $\beta$ is about 10'.

The crystal were monoclinic.

$\underline{a}$ = 9.59. $\underline{b}$ = 39.04 $\underline{c}$ = 12.13Å.

$\beta = 117^{\circ}08'$.

The conditions limiting the observed reflections are

h0l, l = 2n.

0k0, k = 2n.

Space group $P2_1/c$.

dm = 1.39 g. $cm^{-3}$.

With 20 units of $CH_3Zn\,S\,C\,(CH_3)_3$ per unit cell the calculated value of the density is 1.382 g. $cm^{-3}$.

Linear absorption coefficient, Cu. K$\alpha$ radiation = 58.04 $cm^{-1}$

## Data Collection and Correction

The crystal used in the collection of the intensity data was a needle with parallel faces and about 3mm. in length. It had an eight-sided cross-section which was in the form of a rectangle with each corner replaced by an additional face. The ends of the needle were not well developed.

The 0kl, 1kl, 2kl, 3kl, 4kl, 5kl, and 6kl reciprocal lattice nets were recorded photographically using the multifilm equi-inclination Weissenberg technique and Cu. K$\alpha$ radiation. At the end of the data collection it was noted that the crystal,which was originally transparent,had aquired a milky appearance. There was, however, no noticeable deterioration in the quality of the data. The intensities of reflections were estimated visually by comparision with a graduated scale. The intensities were placed on a common scale using intensity measurements derived from photographs on which timed exposures of different nets had been made.

The intensities were corrected for anisotropic absorption and for Lorentz and polarisation factors. In the case of upper layers the intensities were also corrected for spot length (Phillips, 1956).

As the whole of the length of the crystal was not bathed in the X-ray beam the absorption correction was computed by dividing the cross-sectional area of the crystal into 18 elements. The transmission coefficient was then determined by carrying out a summation over two dimensions. The crystal faces were represented by the 10 inequalities given below. The faces were referred to three orthogonal axes, X, Y and Z where $\underline{b}^*$ lies along X, $c^*$ along Y and $\underline{a}$, the rotation axis, along Z.

$$
\begin{array}{rrcrcl}
 & -y & + & 0.080 & \geq & 0 \\
 & y & + & 0.080 & \geq & 0 \\
 & -x & + & 0.175 & \geq & 0 \\
 & x & + & 0.175 & \geq & 0 \\
 & -z & + & 1.500 & \geq & 0 \\
 & z & + & 1.500 & \geq & 0 \\
-0.558\,x & -y & + & 0.108 & \geq & 0 \\
0.558\,x & +y & + & 0.108 & \geq & 0 \\
0.558\,x & -y & + & 0.108 & \geq & 0 \\
-0.558\,x & +y & + & 0.108 & \geq & 0
\end{array}
$$

The lengths are expressed in millimetres.

The exact values of the constants in the inequalities involving z are not important, as faces represented by these inequalities are included only because the programme requires that the crystal shape should be a closed solid.

## Structure Determination

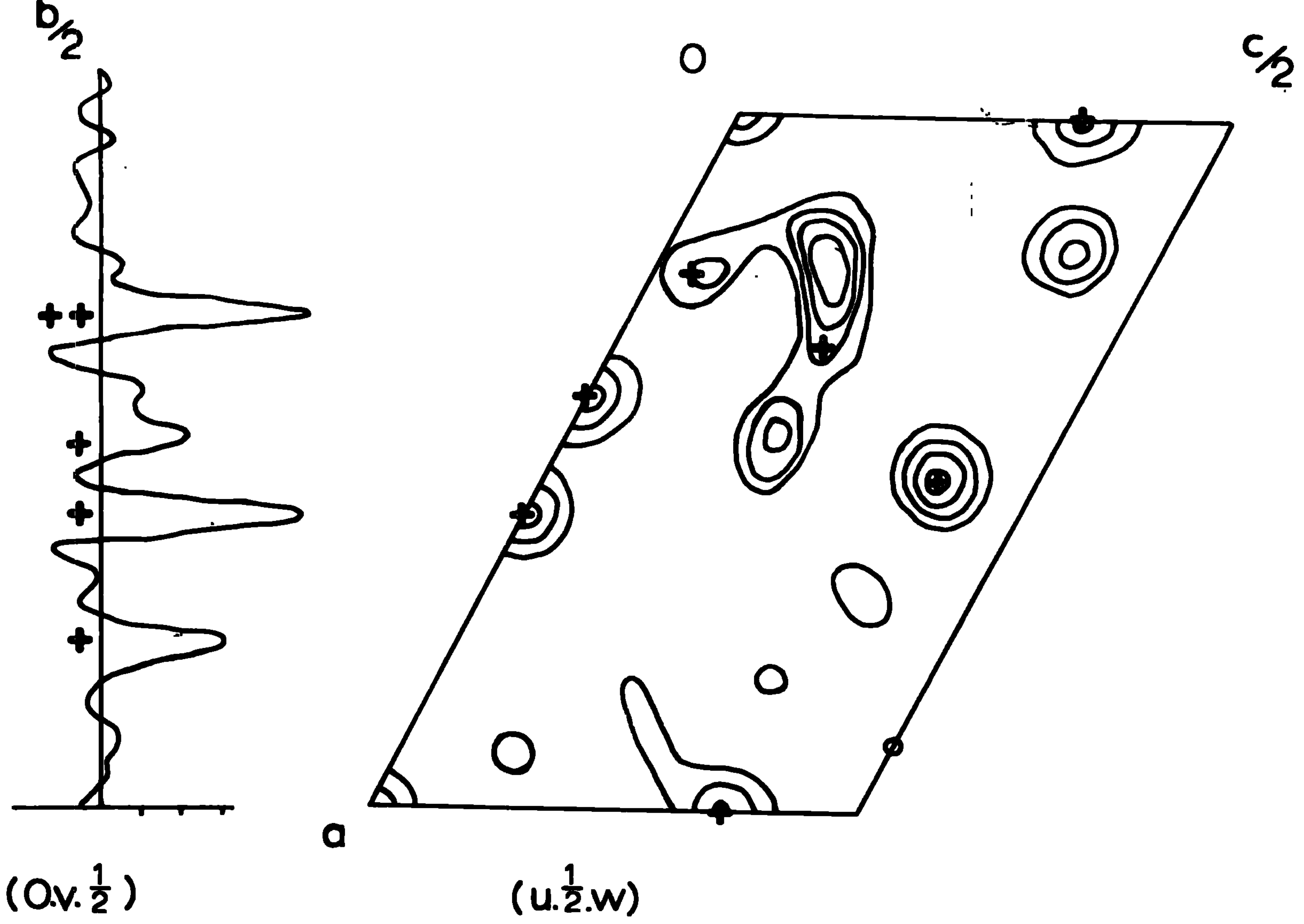
The intensities were multiplied by a weighting function w, where $w = \exp\left(\frac{12 \sin^2 \theta}{\lambda^2}\right)$ The weighting function was not allowed to take a value greater than 100. The Patterson function was calculated using the weighted intensities as coefficients.

The examination of the Patterson function was carried out in order to determine the positions of five zinc atoms in the asymmetric unit. The Harker sections are shown in Fig.1. On the Harker section at (u,½,w)., where peaks due to vectors between atoms related by $2_1$ screw axes occur, there were eight peaks of a sufficient height to represent zinc-zinc vectors of this type. On the Harker section (o,v,½), where peaks due to atoms related by the axial glide plane occur, there were four peaks large enough to be due to zinc-zinc double-weight vectors. Two of these peaks were large enough to arise from the coincidence of such vectors. From the possible occurrence of multiple peaks along (o,½,w) and of extra peaks on (u,½,w) it was deduced that, in the unit cell, zinc atoms which were unrelated by symmetry might have similar y-coordinates.

The positions of Zn2, Zn3 and Zn5 were found from this examination of the Patterson function. These positions were confirmed by finding suitable peaks on the Patterson function, corresponding to vectors between Zn2, Zn3 and Zn5 and the atoms

$(MeZnSBu^t)_5$

**Fig.1. Harker sections (contours are at equal, arbitrary, intervals).**
+ Shows the position of a Zn-Zn double-weight vector.



related to them by the symmetry operations of the space group. Structure factors were calculated, based on the parameters of these three atoms, giving a residual of 0.67.

The positions of Zn1 and Zn4 were found from a superposition of the Patterson function on the single-weight peak due to the vector between Zn3 and the atom related to it by the centre of symmetry at the origin. From this superposition 23 regions of overlap resulted. All but two of these regions were eliminated by placing in turn a zinc atom in each of these regions of overlap, calculating the vectors which this atom would give with symmetry related atoms and checking the Patterson function for suitable peaks. Zn1 and Zn4 were placed at the remaining two regions of overlap.

In order to check the correctness of this solution, the positions of all peaks due to Zn-Zn vectors were calculated and checked with the Patterson function. No attempt was made to determine the positions of the sulphur atoms at this stage.

Structure factors were calculated, based on the five zinc atoms, giving a residual of 0.543. An electron density map was then computed and this allowed the positions of the five sulphur atoms, S1 to S5, to be found. Several small peaks were observed. Twenty five of these peaks could be interpreted as being due to the carbon atoms of the molecule.

The interatomic distances and angles involving the carbon atoms were in poor agreement with the accepted values and the heights of the peaks due to these atoms were low. For these reasons positions were not given to the carbon atoms at this stage.

The positional and isotropic thermal parameters of the zinc and sulphur atoms were then refined by two cycles of least squares calculations. This produced a residual of 0.360. Another Fo synthesis was computed and from this, positions were given to the carbon atoms, C1 to C5, bonded to the zinc atoms, and to the carbon atoms, C6 to C25, of the t-butyl groups bonded to sulphur. The heights of the peaks due to the carbon atoms ranged from about 1.9 to 4.2 e.$\text{Å}^{-3}$.

The parameters of the zinc, sulphur and carbon atoms were refined by the method of least squares using the block diagonal approximation. One cycle of least squares refinement was computed with isotropic temperature parameters, followed by six cycles in which the zinc and sulphur atoms were given anisotropic thermal parameters. This reduced the residual to 0.170. Five cycles of refinement with anisotropic temperature parameters for the carbon atoms also, further reduced the residual to 0.138.

Up to this point in the refinement the bond lengths and angles in the t-butyl group which is bonded to S3 differed considerably from the accepted values. An Fo synthesis was calculated. This showed that the peaks due to the methyl carbon atoms, C15, C16 and C17, of this t-butyl group were broad and that their heights were low at about 2.1 e.Å$^{-3}$.

Three cycles of least squares refinement were then computed using anisotropic temperature factors for the zinc, sulphur and carbon atoms and with refinement of the individual scale factors of the reciprocal lattice nets okl to 6kl. This treatment would be expected to lead to a high correlation between individual scale factors and the anisotropic vibration tensor element $U_{11}$, as the data were collected in nets up the a axis. In this case quite accurate values of the individual scale factors were obtained experimentally and the refinement of the structure was almost complete before the refinement of the individual scale factors was started. The scale factors changed very little during this process, the mean difference between the observed and refined values being 2.7%.

The final value of the residual, calculated over the observed reflections only, was 0.136.

## Final cycle of refinement

$$\sum |Fo| = 101011, \quad \sum |Fc| = 95500, \sum \Delta = 13745, \quad R = 0.136$$

$$\sum w|Fo|^2 = 437.4, \quad \sum w|Fc|^2 = 421.4, \sum \omega \Delta^2 = 17.7, \quad R' = 0.040$$

Number of independent reflections = 2230.

On the final cycle of refinement the largest parameter shift was 0.61 of its e.s.d. The structure factors were weighted by a function $\sqrt{w}$ where,

$$\sqrt{w} = 1/ \left(300 + |Fo| + 0.1\,|Fo|^2\right)^{\frac{1}{2}}$$

Unobserved reflections were given zero weight in the refinement. The atomic scattering factors used were those quoted in 'International Tables for X-ray Crystallography' (1962) Vol III. The real part of the dispersion correction was applied in the case of zinc and sulphur.

The final values of the positional and anisotropic thermal parameters together with their e.s.d.'s are quoted in Tables 1 and 2. The final values of the structure factors are quoted in Table 8.

## Final Difference Map

An electron density difference synthesis was computed using the structure factors obtained from the final cycle of refinement.

This showed a peak of height about 0.5 e.Å$^{-3}$ in the vicinity of C17, one of the atoms which gave rise to a broad peak in the previous Fo synthesis. An examination of the electron density in the region of the atoms C14, C15, C16 and C17 of the t-butyl group bonded to S3 did not give any indication of disorder in the positions of the methyl carbon atoms, or of free rotation about the S3 - C14 bond. Outside this region there were no pronounced features near any of the atomic sites.

## Description of Structure.

The bond lengths and angles with their e.s.d.'s are shown in Table 4 and Table 5 respectively. The correlation coefficients $a_{13}$ shown in Table 3 were included in the calculation of the e.s.d.'s. Some non-bonding intramolecular distances are shown in Table 6, and the non-bonding intermolecular distances less than 4Å are shown in Table 7.

In the crystalline state, methylzinc t-butylsulphide exists as discrete pentameric molecules. The zinc and sulphur atoms are in a polyhedral arrangement, as shown in Fig 2, which also shows the bond lengths involving the zinc and sulphur atoms. The numbering of the atoms is shown in Fig.3. The zinc atoms lie near the corners of a square-based pyramid. The apical zinc atom, Zn5, is displaced from the position above the centre of the base such that it is closer to Zn3 and Zn4 than to Zn1 and Zn2. One sulphur atom lies above each of the faces of the pyramid formed by the zinc atoms. S1, which lies above the face formed by Zn1, Zn2 and Zn5, is much closer to Zn1 and Zn2, and is three-coordinate. S5, which lies below the basal plane of the figure formed by the zinc atoms, seems to be five-coordinate. All of the zinc atoms and S2, S3 and S4 are four-coordinate.

The atoms Zn1, Zn2, Zn3, and Zn4, which form the base of the pyramid are almost coplanar. The equation of the mean plane of the atoms is.

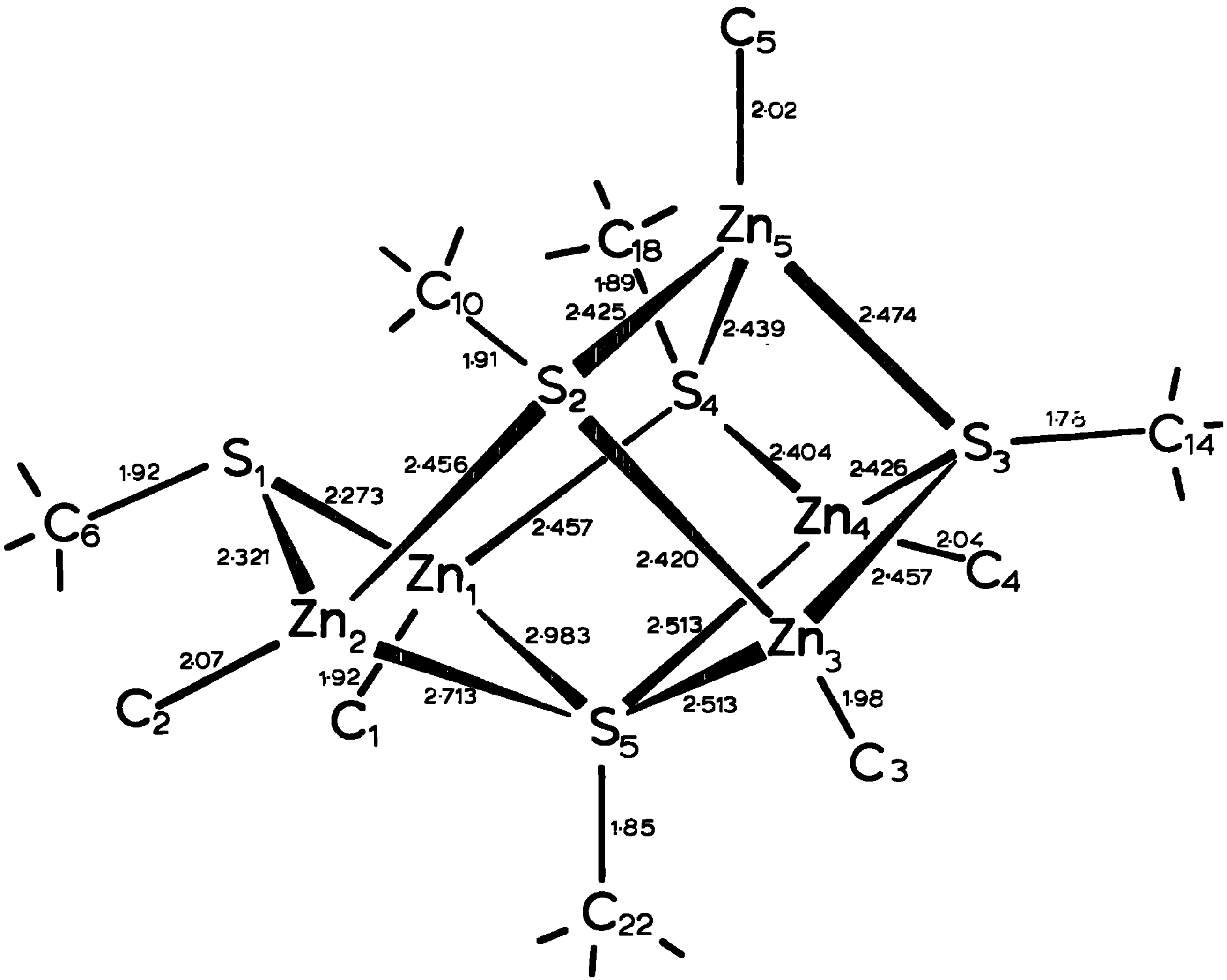
$$0.6762x' + 0.4568y - 0.5780z' + 4.396 = 0$$

The equation is referred to an orthogal coordinate system, in which c' is perpendicular to a and b. All of the atoms lie 0.03 Å from the plane. The distance around the perimeter of the base range from 3.387 to 3.492 Å, individual values being shown in Table 6. The angles are Zn1-Zn2-Zn3 = $94.5^{\circ}$, Zn2-Zn3-Zn4 = $86.8^{\circ}$, Zn3-Zn4-Zn1 = $91.5^{\circ}$ and Zn4-Zn1-Zn2 = $87.1^{\circ}$. The shape of the base of the pyramid deviates significantly from a square.

Zn5 lies above this plane, and is closer to Zn3(3.514 Å) and Zn4(3.511 Å) than to Zn1(4.418 Å) and to Zn2(4.343 Å). The shortest zinc-zinc distance is between Zn1 and Zn2, and is 3.387 Å. This is greater than the distances of 2.665 and 2.913 Å found in metallic zinc (International Tables, 1962) and shows that there is no appreciable zinc-zinc bonding.

One sulphur atom lies above each face of the pyramid formed by the zinc atoms, the shortest sulphur-sulphur distance being 3.34 Å for S2-S5. The single bond tetrahedral covalent radius of sulphur is 1.04 Å (Pauling, 1960) and so there is no appreciable sulphur-sulphur bonding in the molecule.

S1 lies above the plane of Zn1, Zn2 and Zn5 but is much closer to Zn1 (2.273 ± 0.009 Å) and Zn2 (2.321 ± 0.009 Å) than to Zn5 (4.503 Å). Thus S1 is bonded to Zn1 and Zn2 but not to Zn5 and so is three-coordinate. The bond angles at S1 are Zn1 - S1 - Zn2 = $95.0 \pm 0.3^{\circ}$, Zn1 - S1 - C6 = $110.6 \pm 1.1^{\circ}$ and Zn2- S1- C6 = $107.7 \pm 1.1^{\circ}$.


C5
2·02
Zn5
C18
1·89
C10
2·425
2·439
2·474
1·91
S2
S4
1·76
C14
S3
S1
1·92
2·456
2·404
2·426
C6
2·273
Zn4
2·457
2·04
2·321
Zn1
2·420
C4
2·457
Zn2
2·983
2·513
Zn3
2·07
1·92
2·713
2·513
1·98
C2
C1
S5
C3
1·85
C22

$(MeZnSBu^{t})_5$

Fig.2. Zinc-sulphur polyhedron.

## $(MeZnSBu^t)_5$. TABLE 1

### Final Values of Atomic Coordinates and their Standard Deviations.

| Atom | $^x/a$ | $^y/b$ | $^z/c$ | $\sigma(^x/a)$ | $\sigma(^y/b)$ | $\sigma(^z/c)$ |
|---|---|---|---|---|---|---|
| Zn1 | 0·10706 | 0·30608 | 0·27402 | 0·00053 | 0·00010 | 0·00042 |
| Zn2 | 0·23773 | 0·34877 | 0·54613 | 0·00049 | 0·00010 | 0·00035 |
| Zn3 | -0·00701 | 0·41712 | 0·42125 | 0·00047 | 0·00010 | 0·00038 |
| Zn4 | -0·15716 | 0·37329 | 0·14044 | 0·00045 | 0·00012 | 0·00036 |
| Zn5 | 0·20423 | 0·41563 | 0·24886 | 0·00052 | 0·00010 | 0·00041 |
| | | | | | | |
| S1 | 0·33983 | 0·31400 | 0·44525 | 0·00086 | 0·00018 | 0·00075 |
| S2 | 0·25904 | 0·40457 | 0·46164 | 0·00079 | 0·00018 | 0·00061 |
| S3 | -0·06528 | 0·43117 | 0·20711 | 0·00085 | 0·00019 | 0·00071 |
| S4 | 0·09898 | 0·35942 | 0·16381 | 0·00083 | 0·00018 | 0·00066 |
| S5 | -0·05923 | 0·35500 | 0·36211 | 0·00078 | 0·00017 | 0·00061 |
| | | | | | | |
| C1 | -0·0228 | 0·2675 | 0·1932 | 0·0046 | 0·0008 | 0·0032 |
| C2 | 0·2440 | 0·3441 | 0·7183 | 0·0035 | 0·0009 | 0·0026 |
| C3 | -0·0803 | 0·4433 | 0·5250 | 0·0038 | 0·0008 | 0·0032 |
| C4 | -0·3654 | 0·3588 | -0·0032 | 0·0034 | 0·0011 | 0·0031 |
| C5 | 0·3340 | 0·4454 | 0·1943 | 0·0049 | 0·0010 | 0·0040 |
| C6 | 0·4098 | 0·2722 | 0·5384 | 0·0041 | 0·0007 | 0·0036 |
| C7 | 0·5466 | 0·2842 | 0·6631 | 0·0043 | 0·0010 | 0·0036 |
| C8 | 0·2702 | 0·2614 | 0·5732 | 0·0048 | 0·0008 | 0·0038 |
| C9 | 0·4327 | 0·2465 | 0·4621 | 0·0038 | 0·0007 | 0·0037 |
| C10 | 0·3950 | 0·4382 | 0·5724 | 0·0034 | 0·0009 | 0·0034 |
| C11 | 0·3218 | 0·4714 | 0·5147 | 0·0054 | 0·0008 | 0·0043 |
| C12 | 0·3935 | 0·4310 | 0·6985 | 0·0040 | 0·0010 | 0·0032 |
| C13 | 0·5632 | 0·4293 | 0·5927 | 0·0047 | 0·0015 | 0·0042 |
| C14 | -0·1713 | 0·4689 | 0·1309 | 0·0050 | 0·0008 | 0·0035 |
| C15 | -0·0611 | 0·4981 | 0·1792 | 0·0080 | 0·0011 | 0·0055 |

TABLE 1 (Cont.)

| | | | | | | |
|---|---|---|---|---|---|---|
| C16 | -0·2030 | 0·4655 | -0·0115 | 0·0088 | 0·0009 | 0·0046 |
| C17 | -0·3482 | 0·4684 | 0·1451 | 0·0084 | 0·0020 | 0·0071 |
| C18 | 0·0887 | 0·3555 | 0·0052 | 0·0039 | 0·0008 | 0·0022 |
| C19 | 0·0274 | 0·3866 | -0·0643 | 0·0050 | 0·0011 | 0·0036 |
| C20 | 0·2622 | 0·3463 | 0·0209 | 0·0043 | 0·0010 | 0·0037 |
| C21 | -0·0193 | 0·3212 | -0·0616 | 0·0042 | 0·0010 | 0·0030 |
| C22 | -0·2223 | 0·3404 | 0·3896 | 0·0034 | 0·0010 | 0·0035 |
| C23 | -0·2971 | 0·3065 | 0·3146 | 0·0068 | 0·0015 | 0·0059 |
| C24 | -0·3522 | 0·3681 | 0·3443 | 0·0041 | 0·0010 | 0·0033 |
| C25 | -0·1695 | 0·3406 | 0·5292 | 0·0042 | 0·0009 | 0·0030 |

## $(MeZnSBu^t)_5$. TABLE 2

## Final Values of Anisotropic Temperature Parameters and their Standard Deviations.

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $2U_{23}$ | $2U_{31}$ | $2U_{12}$ |
|---|---|---|---|---|---|---|
| Zn1 | 0·1080(34) | 0·1004(28) | 0·1064(32) | 0·0034(48) | 0·0801(53) | −0·0262(47) |
| Zn2 | 0·1086(34) | 0·0970(26) | 0·0877(27) | 0·0216(42) | 0·1022(49) | 0·0267(45) |
| Zn3 | 0·0820(30) | 0·1014(28) | 0·1107(31) | −0·0386(46) | 0·1023(48) | −0·0066(42) |
| Zn4 | 0·0670(28) | 0·1424(35) | 0·0854(27) | −0·0053(50) | 0·0546(42) | −0·0081(48) |
| Zn5 | 0·1004(32) | 0·1074(29) | 0·1073(30) | 0·0223(48) | 0·1076(51) | −0·0254(47) |
| S1 | 0·0739(54) | 0·0900(49) | 0·1125(60) | 0·0375(86) | 0·0935(90) | 0·0203(77) |
| S2 | 0·0713(51) | 0·0853(44) | 0·0734(45) | −0·0019(70) | 0·0516(75) | −0·0129(69) |
| S3 | 0·0711(53) | 0·1008(52) | 0·0948(53) | 0·0377(85) | 0·0615(81) | 0·0274(81) |
| S4 | 0·0720(51) | 0·0906(46) | 0·0832(49) | 0·0071(73) | 0·0734(79) | 0·0103(71) |
| S5 | 0·0645(47) | 0·0839(42) | 0·0730(43) | 0·0028(69) | 0·0644(73) | −0·0123(69) |
| C1 | 0·178(35) | 0·102(22) | 0·137(28) | −0·092(41) | 0·204(54) | −0·112(46) |
| C2 | 0·076(22) | 0·168(32) | 0·073(20) | 0·035(39) | 0·043(33) | −0·026(39) |
| C3 | 0·097(26) | 0·130(26) | 0·142(28) | −0·127(44) | 0·141(45) | −0·020(39) |
| C4 | 0·040(19) | 0·274(48) | 0·097(24) | −0·093(56) | 0·020(34) | 0·067(47) |
| C5 | 0·165(38) | 0·135(31) | 0·185(38) | −0·090(56) | 0·195(65) | −0·167(54) |
| C6 | 0·144(30) | 0·061(17) | 0·214(37) | 0·148(41) | 0·225(56) | 0·109(36) |
| C7 | 0·121(31) | 0·162(34) | 0·134(31) | 0·143(54) | 0·530(48) | 0·083(50) |
| C8 | 0·209(40) | 0·087(21) | 0·226(41) | 0·056(46) | 0·362(73) | −0·033(46) |
| C9 | 0·099(27) | 0·082(20) | 0·227(40) | 0·052(45) | 0·177(53) | 0·115(36) |
| C10 | 0·048(21) | 0·159(30) | 0·154(31) | −0·109(50) | 0·049(38) | −0·043(38) |
| C11 | 0·211(44) | 0·061(19) | 0·209(41) | −0·081(46) | 0·115(68) | 0·080(47) |
| C12 | 0·097(27) | 0·156(31) | 0·101(24) | −0·005(46) | 0·020(39) | −0·067(45) |
| C13 | 0·088(31) | 0·333(66) | 0·153(38) | −0·005(82) | 0·119(55) | 0·015(70) |
| C14 | 0·201(41) | 0·090(23) | 0·125(30) | 0·055(41) | 0·127(58) | 0·001(46) |
| C15 | 0·315(74) | 0·118(33) | 0·230(55) | 0·006(72) | 0·001(101) | 0·133(79) |

TABLE 2 (Cont.)

| | | | | | | |
|---|---|---|---|---|---|---|
| C16 | 0·473(102) | 0·056(21) | 0·161(42) | 0·019(47) | -0·030(105) | -0·028(72) |
| C17 | 0·291(74) | 0·402(93) | 0·356(88) | 0·434(152) | 0·519(145) | 0·486(139) |
| C18 | 0·138(27) | 0·130(23) | 0·039(15) | 0·012(30) | 0·092(34) | 0·003(40) |
| C19 | 0·152(37) | 0·172(37) | 0·122(31) | -0·003(52) | 0·120(56) | 0·056(53) |
| C20 | 0·110(29) | 0·149(31) | 0·155(33) | 0·042(49) | 0·161(52) | 0·063(44) |
| C21 | 0·133(30) | 0·182(36) | 0·083(23) | 0·028(45) | 0·090(43) | 0·059(52) |
| C22 | 0·050(22) | 0·170(32) | 0·176(34) | 0·127(52) | 0·137(44) | 0·015(39) |
| C23 | 0·248(59) | 0·262(59) | 0·309(70) | -0·384(109) | 0·423(116) | -0·340(98) |
| C24 | 0·101(28) | 0·176(36) | 0·119(28) | -0·054(51) | 0·085(44) | 0·014(48) |
| C25 | 0·150(32) | 0·147(28) | 0·108(23) | 0·004(41) | 0·201(47) | -0·046(46) |

The temperature parameters are in units of $Å^2$. The standard deviations of the zinc and sulphur atoms are in $Å^2 \times 10^4$ and those of the carbon atoms in $Å^2 \times 10^3$.

## $(MeZnSBu^t)_5$. TABLE 3

### Correlation Coefficients.

| | $a_{12}$ | $a_{13}$ | $a_{23}$ |
|---|---|---|---|
| Zn1 | -0·08312 | 0·40824 | 0·01041 |
| Zn2 | 0·08615 | 0·49117 | 0·06576 |
| Zn3 | -0·01790 | 0·49684 | -0·12522 |
| Zn4 | -0·04321 | 0·36851 | -0·02875 |
| Zn5 | -0·08074 | 0·49336 | 0·06804 |
| S1 | 0·07321 | 0·46438 | 0·11490 |
| S2 | -0·05460 | 0·37538 | -0·00170 |
| S3 | 0·10016 | 0·40456 | 0·12575 |
| S4 | 0·04862 | 0·45292 | 0·02333 |
| S5 | -0·04382 | 0·44061 | 0·01291 |
| C1 | -0·26716 | 0·57577 | -0·23832 |
| C2 | -0·09309 | 0·34431 | 0·09371 |
| C3 | -0·05538 | 0·52918 | -0·31760 |
| C4 | 0·17027 | 0·26809 | -0·17974 |
| C5 | -0·38259 | 0·53819 | -0·21059 |
| C6 | 0·32175 | 0·55540 | 0·37694 |
| C7 | 0·18991 | 0·26659 | 0·30909 |
| C8 | -0·08513 | 0·70781 | 0·13070 |
| C9 | 0·34558 | 0·50924 | 0·11193 |
| C10 | -0·11319 | 0·30132 | -0·21253 |
| C11 | 0·19337 | 0·34043 | -0·21414 |
| C12 | -0·17715 | 0·24581 | 0·00118 |
| C13 | 0·01692 | 0·45591 | -0·00832 |
| C14 | -0·00110 | 0·42285 | 0·16128 |
| C15 | 0·26341 | 0·00775 | -0·02230 |
| C16 | -0·07022 | -0·00082 | 0·03620 |
| C17 | 0·60765 | 0·75675 | 0·50707 |
| C18 | 0·01977 | 0·50792 | 0·02751 |
| C19 | 0·13112 | 0·41493 | 0·00505 |
| C20 | 0·17931 | 0·50577 | 0·09741 |
| C21 | 0·13829 | 0·43788 | 0·07899 |
| C22 | 0·00720 | 0·54621 | 0·26702 |
| C23 | -0·56779 | 0·70774 | -0·55795 |
| C24 | 0·04739 | 0·40253 | -0·12586 |
| C25 | -0·09530 | 0·66158 | 0·02378 |

## $(MeZnSBu^t)_5$. TABLE 4

## Bond Lengths (Å) and their Standard Deviations (Å x $10^3$)

| | | | |
|---|---|---|---|
| Zn1-S1 | 2·273(9) | S1-C6 | 1·918(30) |
| Zn1-S4 | 2·457(8) | S2-C10 | 1·906(35) |
| Zn1-S5 | 2·982(8) | S3-C14 | 1·783(36) |
| Zn2-S1 | 2·321(9) | S4-C18 | 1·888(24) |
| Zn2-S2 | 2·456(8) | S5-C22 | 1·845(33) |
| Zn2-S5 | 2·713(8) | | |
| Zn3-S2 | 2·420(8) | C6-C7 | 1·566(53) |
| Zn3-S3 | 2·457(9) | C6-C8 | 1·625(52) |
| Zn3-S5 | 2·513(8) | C6-C9 | 1·451(47) |
| Zn4-S3 | 2·426(9) | C10-C11 | 1·482(50) |
| Zn4-S4 | 2·404(8) | C10-C12 | 1·571(55) |
| Zn4-S5 | 2·513(8) | C10-C13 | 1·562(52) |
| Zn5-S2 | 2·426(8) | C14-C15 | 1·475(66) |
| Zn5-S3 | 2·474(9) | C14-C16 | 1·619(78) |
| Zn5-S4 | 2·439(8) | C14-C17 | 1·784(81) |
| | | C18-C19 | 1·445(51) |
| Zn1-C1 | 1·915(35) | C18-C20 | 1·620(49) |
| Zn2-C2 | 2·072(31) | C18-C21 | 1·666(50) |
| Zn3-C3 | 1·982(33) | C22-C23 | 1·599(67) |
| Zn4-C4 | 2·043(32) | C22-C24 | 1·533(50) |
| Zn5-C5 | 2·023(41) | C22-C25 | 1·522(46) |

## $(MeZnSBu^t)_5$. TABLE 5

## Bond Angles with their Standard Deviations

| Atoms | Angle | e.s.d. |
|---|---|---|
| S1-Zn1-S4 | 99·0° | 0·3° |
| S1-Zn1-S5 | 92·8 | 0·3 |
| S4-Zn1-S5 | 76·3 | 0·3 |
| S1-Zn1-C1 | 135·1 | 1·1 |
| S4-Zn1-C1 | 122·4 | 1·0 |
| S5-Zn1-C1 | 111·1 | 1·1 |
| S1-Zn2-S2 | 99·4 | 0·3 |
| S1-Zn2-S5 | 99·0 | 0·3 |
| S2-Zn2-S5 | 80·4 | 0·2 |
| S1-Zn2-C2 | 131·0 | 1·0 |
| S2-Zn2-C2 | 122·0 | 1·0 |
| S5-Zn2-C2 | 112·0 | 0·8 |
| S2-Zn3-S3 | 88·2 | 0·3 |
| S2-Zn3-S5 | 85·3 | 0·3 |
| S3-Zn3-S5 | 89·6 | 0·3 |
| S2-Zn3-C3 | 128·5 | 1·0 |
| S3-Zn3-C3 | 125·4 | 1·0 |
| S5-Zn3-C3 | 126·7 | 1·0 |
| S3-Zn4-S4 | 88·5 | 0·3 |
| S3-Zn4-S5 | 90·4 | 0·3 |
| S4-Zn4-S5 | 86·9 | 0·3 |
| S3-Zn4-C4 | 127·3 | 1·2 |
| S4-Zn4-C4 | 125·9 | 1·1 |
| S5-Zn4-C4 | 125·5 | 1·1 |
| S2-Zn5-S3 | 87·7 | 0·3 |
| S2-Zn5-S4 | 97·9 | 0·3 |
| S3-Zn5-S4 | 86·6 | 0·3 |
| S2-Zn5-C5 | 125·5 | 1·2 |
| S3-Zn5-C5 | 122·6 | 1·2 |
| S4-Zn5-C5 | 125·2 | 1·2 |
| Zn1-S1-Zn2 | 95·0 | 0·3 |
| Zn1-S1-C6 | 110·6 | 1·1 |
| Zn2-S1-C6 | 107·7 | 1·1 |
| Zn2-S2-Zn5 | 126·7 | 0·3 |
| Zn2-S2-Zn3 | 89·0 | 0·3 |
| Zn3-S2-Zn5 | 93·0 | 0·3 |
| Zn2-S2-C10 | 118·7 | 1·2 |
| Zn3-S2-C10 | 108·9 | 1·0 |
| Zn5-S2-C10 | 111·8 | 1·2 |
| Zn3-S3-Zn4 | 91·0 | 0·3 |
| Zn3-S3-Zn5 | 91·0 | 0·3 |
| Zn4-S3-Zn5 | 91·5 | 0·3 |
| Zn3-S3-C14 | 122·1 | 1·3 |
| Zn4-S3-C14 | 124·2 | 1·3 |

TABLE 5 (cont.)

| | | |
|---|---|---|
| Zn5-S3-C14 | 127·0 | 1·4 |
| Zn1-S4-Zn4 | 91·8 | 0·3 |
| Zn1-S4-Zn5 | 128·9 | 0·3 |
| Zn4-S4-Zn5 | 93·0 | 0·3 |
| Zn1-S4-C18 | 117·4 | 1·0 |
| Zn4-S4-C18 | 109·0 | 1·1 |
| Zn5-S4-C18 | 108·8 | 1·0 |
| Zn1-S5-Zn2 | 72·8 | 0·2 |
| Zn1-S5-Zn3 | 130·6 | 0·3 |
| Zn1-S5-Zn4 | 78·4 | 0·2 |
| Zn2-S5-Zn3 | 81·6 | 0·2 |
| Zn2-S5-Zn4 | 130·2 | 0·3 |
| Zn3-S5-Zn4 | 87·7 | 0·3 |
| Zn1-S5-C22 | 120·7 | 1·2 |
| Zn2-S5-C22 | 118·9 | 1·1 |
| Zn3-S5-C22 | 108·6 | 1·2 |
| Zn4-S5-C22 | 110·6 | 1·1 |
| S1-C6-C7 | 103·0 | 2·0 |
| S1-C6-C8 | 105·6 | 2·1 |
| S1-C6-C9 | 109·2 | 2·5 |
| C7-C6-C8 | 104·4 | 2·9 |
| C7-C6-C9 | 121·3 | 3·0 |
| C8-C6-C9 | 112·0 | 2·5 |
| S2-C10-C11 | 104·4 | 2·4 |
| S2-C10-C12 | 104·6 | 2·3 |
| S2-C10-C13 | 106·3 | 2·8 |
| C11-C10-C12 | 114·1 | 3·3 |
| C11-C10-C13 | 120·4 | 3·6 |
| C12-C10-C13 | 105·8 | 2·9 |
| S3-C14-C15 | 107·5 | 3·3 |
| S3-C14-C16 | 105·7 | 2·6 |
| S3-C14-C17 | 106·3 | 3·0 |
| C15-C14-C16 | 104·1 | 4·1 |
| C15-C14-C17 | 120·4 | 4·2 |
| C16-C14-C17 | 112·0 | 4·1 |
| S4-C18-C19 | 110·3 | 2·3 |
| S4-C18-C20 | 108·3 | 2·1 |
| S4-C18-C21 | 107·0 | 1·8 |
| C19-C18-C20 | 112·4 | 2·8 |
| C19-C18-C21 | 112·9 | 2·8 |
| C20-C18-C21 | 105·0 | 2·4 |
| S5-C22-C23 | 110·1 | 2·8 |
| S5-C22-C24 | 109·2 | 2·5 |
| S5-C22-C25 | 108·7 | 2·2 |
| C23-C22-C24 | 107·3 | 3·1 |
| C23-C22-C25 | 119·1 | 3·3 |
| C24-C22-C25 | 101·9 | 2·8 |

As the environment of S3 is pyramidal, the sulphur atom will be $sp^3$ hybridised, although it is three-coordinate. In a pyramidal molecule the bond angles would be expected to be less than 109°28' because the repulsion between non-bonding electrons and the electrons in the bonding orbitals will be greater than those between the electrons in the different bonding orbitals (Gillespie and Nyholm, 1957). The small value of the Zn1 - S1 - Zn2 angle will lead to some increase in the size of the angles Zn1 - S1 - C6 and Zn2 - S1 - C6.

S2 lies near a point above the centre of the face defined by Zn2, Zn3 and Zn5, S4 is positioned above Zn4, Zn5 and Zn1 and S3 is above Zn3, Zn4 and Zn5. S2, S3 and S4 are four-coordinate. The nine Zn-S bonds formed by these atoms have lengths between 2.404±0.008 Å and 2.474±0.009 Å. S5 lies below the plane of Zn1, Zn2, Zn3, and Zn4, and is closer to Zn3 and Zn4 (both 2.513±0.008Å) than to Zn1 (2.982±0.008 Å) and Zn2 (2.713±0.008 Å).

The seven atoms, Zn3, Zn4, Zn5, S2, S3, S4, and S5 lie near corners of a cube. In addition the molecule has an approximate mirror plane which passes near S1, S3, S5 and Zn5. The equation of the mean plane through S1, S3, S5 and Zn5 is

$$-0.3838x' \quad -0.4799y \quad -0.7890z' \quad -10.1037 = 0$$

The deviation of the atoms from the plane are.

| | |
|---|---|
| S1 | 0.019 Å |
| S3 | 0.033 |
| S5 | -0.023 |
| Zn5 | -0.029 |

The lengths of the Zn-S bonds are related in pairs by the approximate mirror plane. The lengths of pairs of bonds which are related by the approximate mirror plane are listed below, the differences between the lengths of related bonds are expressed as multiples of $\sigma$.

| | | | | |
|---|---|---|---|---|
| Zn1 - S1 | 2.273 Å. | Zn2 - S1 | 2.321 Å. | 3.7 $\sigma$ |
| Zn1 - S5 | 2.982 | Zn2 - S5 | 2.713 | 24.5 |
| Zn1 - S4 | 2.457 | Zn2 - S2 | 2.456 | 0.1 |
| Zn3 - S2 | 2.420 | Zn4 - S4 | 2.404 | 1.5 |
| Zn3 - S3 | 2.457 | Zn4 - S3 | 2.426 | 2.6 |
| Zn3 - S5 | 2.513 | Zn4 - S5 | 2.513 | 0.0 |
| Zn5 - S2 | 2.426 | Zn5 - S4 | 2.439 | 1.2 |

Apart from the lengths Zn1 - S5 and Zn2 - S5 the agreement between members of a pair is very close. The positions of the carbon atoms are also related by the approximate mirror plane.

The outer electronic configuration of zinc is $3d^{10}, 4s^{2}$. In its known ionic compounds zinc does not show an oxidation number greater than two. In compounds in which zinc is regarded as forming covalent bonds it is usually four-coordinate involving the use of 4s and 4p atomic orbitals. Zinc does not use its 'd' orbitals to any appreciable extent in bonding and is not regarded as a transition element.

Sulphur has the outer electronic configuration $3s^2$, $3p^4$. In the sulphides of the most electropositive elements it can be thought of as being present as $S^{2-}$ ions; however, in most of its compounds it forms bonds with considerable covalent character. The Allred - Rochow electronegativities of zinc and sulphur are 1.66 and 2.44 respectively.

In zinc sulphide the zinc and sulphur atoms are four-coordinate with each atom using tetrahedral $sp^3$ hybrid orbitals. If the electrons in the Zn-S bonds were shared equally between the zinc and sulphur atoms, this would lead to the polarity $Zn^{2-} - S^{2+}$. Detailed calculations (Coulson, 1961) suggest that the ionic character of the bonding is such that the zinc atom carries a charge of about +0.33e. The sum of the covalent radii of zinc and sulphur is 2.35 Å (Pauling, 1960), this being equal to the Zn - S distance in zinc sulphide, and in $[(NH_2)_2S]_2\ ZnCl_2$ (Kunchur and Truter, 1958). The sum of the ionic radii of zinc and sulphur is 2.58 Å (Pauling, 1960). The nine Zn - S bonds formed by S2, S3 and S4 have lengths of between 2.404 ± 0.008 and 2.474 ± 0.009 Å. In zinc diethyldithiocarbamate (Bonamico, Mazzone, Vaciago and Zambonelli, 1965) four Zn -S bond lengths range from 2.331 to 2.443 Å and there is one long bond of 2.815 Å. The three sulphur atoms S2, S3 and S4 and the five zinc atoms are four-coordinate. The lengths of the Zn - S bonds formed by S2,S3 and S4 are significantly longer than those in zinc sulphide. The difference can be thought of as arising from a larger ionic contribution to the bonding and, as the bond angles are very close to 90°, from a larger p character in the atomic orbitals used by the zinc and sulphur atoms.

In the methylzinc methoxide tetramer (Shearer and Spencer, 1966) the mean Zn - O distance is 2.09 Å, which again lies between the values of 1.97 and 2.14 Å which would be expected for covalent and ionic bonding respectively (Pauling, 1960).

In $(\mathrm{MeZnSBu^t})_5$, when one pair of electrons has been allocated to each Zn - C and each S - C bond, there remain 30 electrons to bond the zinc-sulphur polyhedron together. If bonds to S5 are excluded, there are 11 Zn - S bonds, to which 22 electrons must be assigned. Two electrons must be placed in the non-bonding $sp^3$ hybrid orbital of S1. There remain 6 electrons to bond S5 to the polyhedron.

To describe the Zn - S bonds involving S5, the 3s and $3p_z$ atomic orbitals are assumed to be hybridised. One sp hybrid orbital is used to form a bond to C22. The other is directed towards the plane of Zn1, Zn2, Zn3 and Zn4. The $3p_x$ and $3p_y$ orbitals of S5 lie in the plane parallel to that of the four zinc atoms. Each of the four zinc atoms will have one atomic orbital which is directed approximately towards S5.

These orbitals of the four zinc atoms can be combined, and of the three combinations of lowest energy, one will have 'a' symmetry and the other two will have '$e_1$' symmetry. The sp hybrid of S5 can interact with the combination of 'a' symmetry and the $3p_x$ and $3p_y$ orbitals have the correct symmetry to interact with the '$e_1$' combination. The six electrons are placed in the resulting three molecular orbitals. Some modification of this treatment would be necessary in order to allow for the variation in the lengths of the bonds between the four zinc atoms and S5.

The Zn3-S5 and Zn4-S5 bond lengths are both 2.513 ± 0.008 Å, and are not very much outside the range of the lengths of the Zn-S bonds formed by S2, S3 and S4. The Zn1-S5 (2.982 ± 0.008 Å) and Zn2-S5 (2.713 ± 0.008 Å) bond lengths are larger than the other Zn-S bonds, and are also greater than the value of 2.58 Å, for the sum of the ionic radii of zinc and sulphur (Pauling, 1960). In zinc diethyldithiocarbamate (Bonamico, Mazzone, Vaciago and Zambonelli, 1965) one Zn-S bond length is 2.815 Å. This indicates that the Zn2-S5 and particularly the Zn1-S5 bonds are very weak. The weakness of the Zn1-S5 and Zn2-S5 bonds is also indicated by the bond angles at Zn1 and Zn2. The sums of the bond angles, excluding bonds to S5, at Zn1 and Zn2 are 356.9° and 352° respectively.

Thus Zn1 and Zn2 are bonded to three atoms with which they are almost coplanar. Zn1 and Zn2 are both displaced from these planes, in the direction of S5.

The bond lengths Zn1-S1 (2.273 ± 0.009 Å) and Zn2-S1 (2.321 ± 0.009 Å) are shorter than the other zinc-sulphur bonds in this compound, and are also shorter then the sum of the tetrahedral covalent radii of zinc and sulphur, which is 2.35 Å (Pauling, 1960). The Zn1-S1 and Zn2-S1 bond lengths would be expected to be short as S1, although it is $sp^3$ hybridised is only three-coordinate. Zn1 and Zn2 interact weakly with S5 and thus have only three near neighbours, so that the use of the tetrahedral radii is not wholly satisfactory. There is also the possibility of some Zn-S $\pi$-bonding involving the non-bonding pair of electrons on S1, and the empty 4d orbitals on Zn1 and Zn2.

The sum of the tetrahedral covalent radii of zinc and carbon (Pauling, 1960) is 2.08 Å. The mean Zn-C bond length found in $(MeZnOMe)_4$ is 1.94 Å (Shearer and Spencer, 1966a). In $Me_2Zn$ (Rundle, 1963) the Zn-C distance is 1.94 Å and the zinc atoms are two-coordinate. A shortening of the Zn-C bond in $Me_2Zn$ was explained by invoking hyperconjugation involving the empty 4p orbitals on the zinc atom. In $(MeZnSBu^t)_5$ the lengths of the Zn-C bonds range from 1.915 ± 0.035 Å for Zn1-C1 to 2.072 ± 0.031 Å for Zn2-C2. The difference in length between Zn1-C1 and Zn2-C2 is $3.3\,\sigma$ and is considered significant.

The Zn2-C2 bond length is not significantly longer than any of the other Zn-C bond lengths. Some difference in the values of the Zn-C bond lengths might be expected in $(MeZnSBu^t)_5$, in view of the different environments of the zinc atoms. The relative shortness of the Zn1-C1 bond length might be related to the fact that the Zn1-S5 interaction is very weak and thus Zn1 can form a stronger bond to C1. The mean Zn-C bond length in $(CH_3ZnSBu^t)_5$ is 2.007 Å.

The S-C bond lengths in this compound vary from S3-C14 (1.783 ± 0.036 Å) to S1-C6 (1.918 ± 0.030 Å). Apart from S3-C14, all of the S-C bonds have lengths which are the same within experimental error. The atom S3 is the only sulphur atom at which there are three Zn-S-Zn bond angles which are very nearly 90°. Thus atomic orbitals which S3 uses to form bonds to the zinc atoms will have a large 'p' character and the atomic orbital used by S3 in the S3-C14 bond will have large 's' character, leading to a short bond. The mean S-C distance is 1.868 Å. The S-C bond length in $CH_3SH$ is 1.815 ± (Kojima and Nishikawa, 1957). Some difference between the S-C bond length in $(MeZnSBu^t)_5$ and that in $CH_3SH$ is would be expected as in $CH_3SH$, sulphur is two coordinate whereas in $(MeZnSBu^t)_5$ sulphur atoms are three-, four - and five - coordinate.

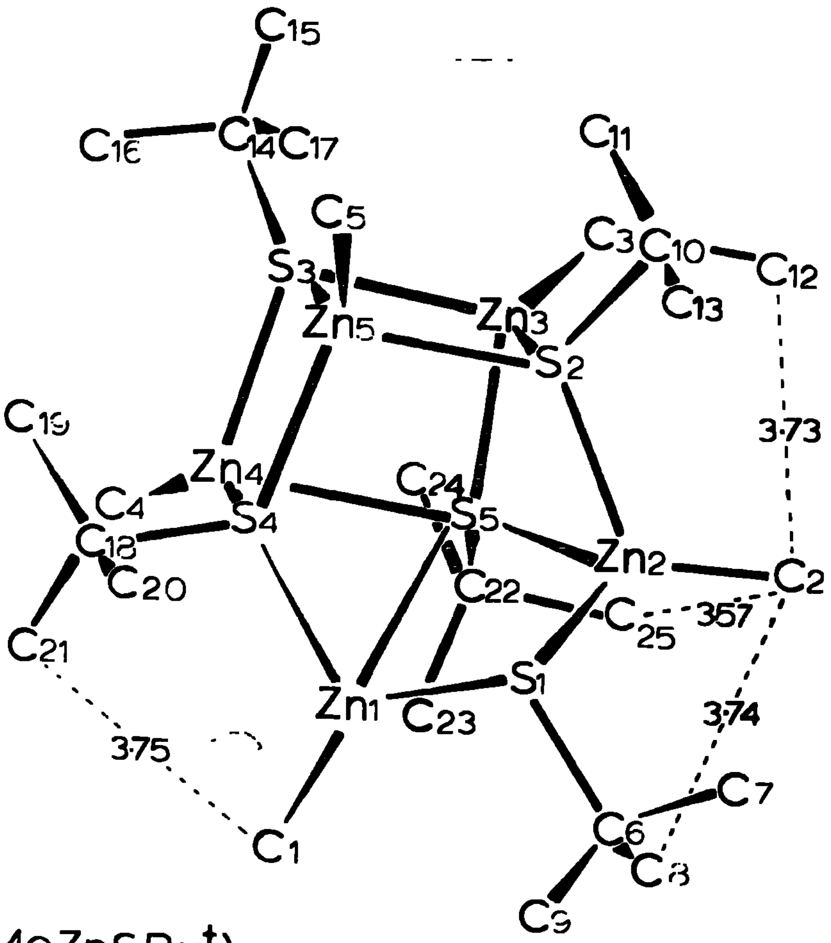
The mean C-C bond length is 1.568 Å which can be compared with the value of 1.5445 Å found in diamond. The C-C bond length which deviates most from that in diamond is C14-C17 (1.784 ± 0.081 Å) where the deviation is $2.95\sigma$. This would normally be considered significant. In the electron density maps, C17 gave rise to peaks which were very much smeared out, and showed that there was appreciable overlap of the electron densities due to C14 and C17. The atomic parameters were refined using the method of least squares with the block diagonal approximation. This refinement results in C17 having very large temperature parameters. Neglect of certain off-diagonal elements in the matrix may lead to underestimates of the e.s.d.'s of the atomic parameters (Hodgson and Rollett, 1963) particularly if there is large overlap of electron density. The correlation coefficients $a_{12}$ and $a_{13}$ which are normally very small, were large in the case of C17. In view of the possible underestimate of the e.s.d.'s it is not considered worthwhile to discuss the length of the C14-C17 bond.

The S-Zn-C and Zn-S-C bond angles are related in pairs by the approximate mirror plane. The Zn-S-C angles at S1 have been discussed earlier. At S5 the angles Zn3-S5-C22 and Zn4-S5-C22 are significantly larger than Zn1-S5-C22 and Zn2-S5-C22. In the case of the C-C-C bond angles, it is noted that in each t-butyl group there is one large C-C-C angle of about 120°, one angle of about 111°, and one of about 104°. Only one angle, at C18 does not appear to fit into this pattern but the difference is $2.5\sigma$. This distortion of the bond angles is such that a methyl group bonded to a zinc atom lies in a direction between those of the two

C-C bonds which have the large angle between them.

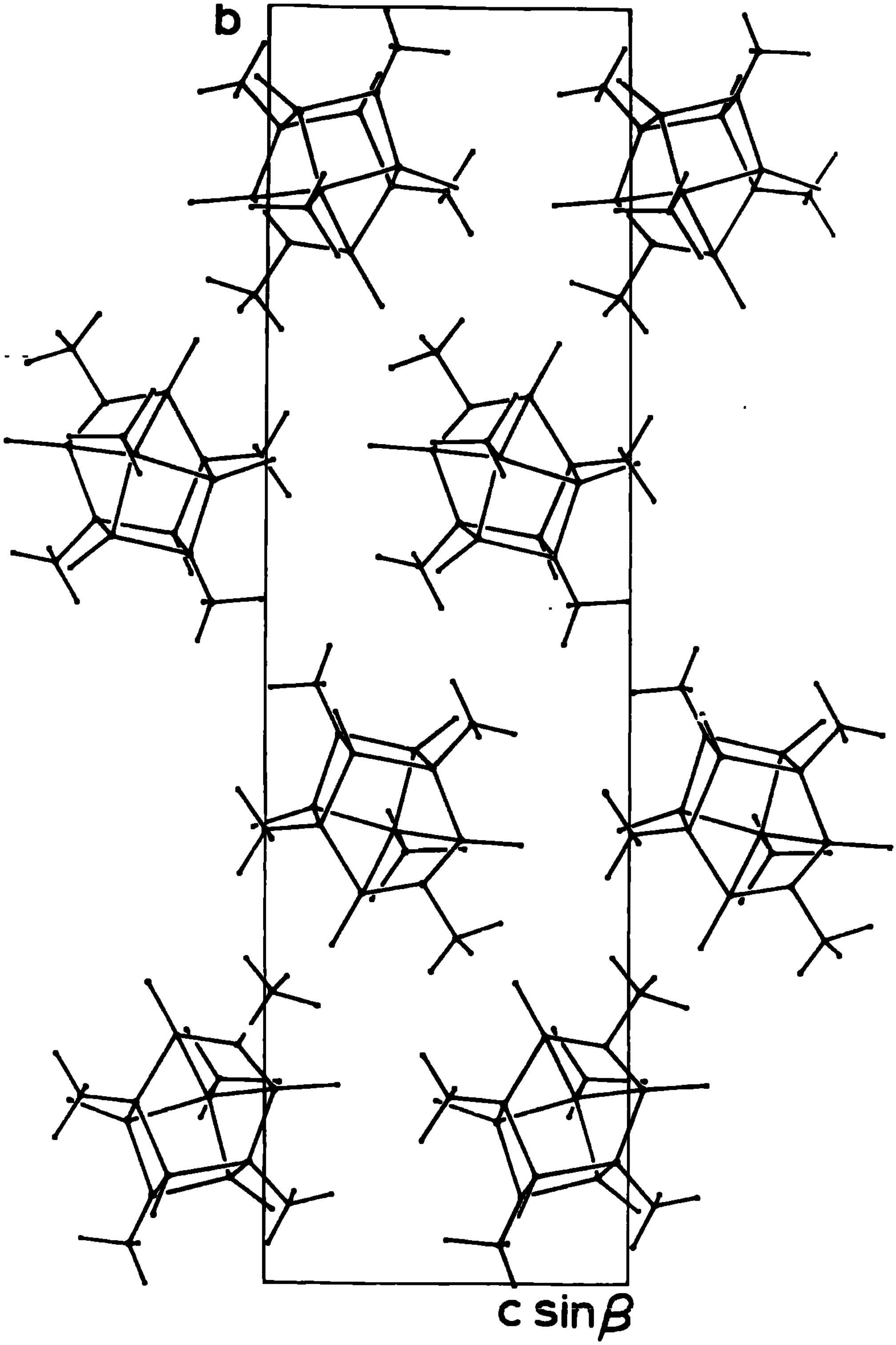
Eight intramolecular non-bonding contacts, and nine intermolecular contacts are less than 4 Å. Four intramolecular contacts and two intermolecular contacts, all involving C2, lie between 3.60 and 3.81 Å. The shortest non-bonding contact is between C5 and C17 in the molecule at (1 + x,y,z) and is 3.48 Å. However the position of C17 may be in error by up to about 0.2 Å. The value of the van der Waals radius of a methyl group is quoted as 2.0 Å (Pauling, 1960). The intramolecular contacts between methyl carbon atoms are comparable with those found between molecules, so that no unusual steric effects are operating.

The temperature parameters of the zinc atoms are larger than those of the sulphur atoms. The form factors used for zinc and sulphur were those of the neutral atoms. In this compound the zinc atoms will carry a small positive charge and the sulphur atoms a small negative charge. The temperature parameters obtained from the refinement will tend to allow for this effect. The temperature factors of all atoms are large, in particular those of carbon atoms are much greater than those normally encountered. The molecules might have large thermal motions due to their approximately spherical shape and because their outer region is made up of alkyl groups. The large thermal parameters might be attributed in part to positional disorder due to some deterioration of the crystal.


C15
C16
C14
C17
C11
C5
C3
C10
C12
S3
Zn5
Zn3
C13
S2
C19
Zn4
C24
3·73
C4
S4
S5
C18
Zn2
C2
C20
C22
C25
3·57
C21
S1
Zn1
C23
3·74
3·75
C7
C6
C1
C8
C9

$(MeZnSBu^t)_5$

Fig. 3.



(MeZnSBu$^t$)$_5$ [100] projection

Fig.4.

## $(MeZnSBu^t)_5$. TABLE 6

### Some Non-bonding Intramolecular Distances (Å)

| | | | |
|---|---|---|---|
| Zn1-Zn2 | 3·387 | C1-C21 | 3·75 |
| Zn1-Zn3 | 4·997 | C2-C23 | 3·81 |
| Zn1-Zn4 | 3·492 | C2-C8 | 3·74 |
| Zn1-Zn5 | 4·418 | C2-C12 | 3·73 |
| Zn2-Zn3 | 3·418 | C2-C25 | 3·57 |
| Zn2-Zn4 | 4·741 | C3-C24 | 3·88 |
| Zn2-Zn5 | 4·343 | C4-C21 | 3·97 |
| Zn3-Zn4 | 3·483 | C5-C19 | 3·92 |
| Zn3-Zn5 | 3·515 | | |
| Zn4-Zn5 | 3·511 | | |
| Zn5-S1 | 4·503 | | |
| S1-S2 | 3·645 | | |
| S1-S3 | 5·842 | | |
| S1-S4 | 3·597 | | |
| S1-S5 | 3·836 | | |
| S2-S3 | 3·396 | | |
| S2-S4 | 3·668 | | |
| S2-S5 | 3·342 | | |
| S3-S4 | 3·370 | | |
| S3-S5 | 3·504 | | |
| S4-S5 | 3·384 | | |

## $(MeZnSBu^t)_5$. TABLE 7

## Non-bonding Intermolecular Contacts (Å)

The position of the second atom is indicated by the operations given in column 2.

| | | |
|---|---|---|
| C1-C8 | $x, \frac{1}{2} - y, -\frac{1}{2} + z$ | 3·87 |
| C2-C20 | $x, y, 1 + z$ | 3·60 |
| C2-C4 | $1 + x, y, 1 + z$ | 3·77 |
| C3-C13 | $-1 + x, y, z$ | 3·90 |
| C3-C15 | $-x, 1 - y, 1 - z$ | 3·94 |
| C3-C11 | $-x, 1 - y, 1 - z$ | 3·96 |
| C4-C20 | $-1 + x, y, z$ | 3·76 |
| C5-C17 | $1 + x, y, z$ | 3·48 |
| C7-C23 | $1 + x, \frac{1}{2} - y, \frac{1}{2} + z$ | 3·95 |

## Discussion

In $B_5H_9$ (Lipscomb, 1963) and $Fe_5(CO)_{15}C$ (Braye, Dahl, Hubel and Wampler, 1962) metal atoms are situated at the corners of a square-based pyramid. The structures of these compounds differ from that of $(MeZnSBu^t)_5$ in that they contain metal-metal bonds, and the molecules are much more symmetrical. In $(MeZnSBu^t)_5$, sulphur is found bridging two, three and four zinc atoms. Many dimeric transition metal complexes containing sulphur bridges are known. In the compound $Fe_2(CO)_6(EtS)_2$ (King, 1963; Dahl and Wei, 1963) the $EtS^-$ ion can be thought of as bridging two iron atoms. There is an Fe-Fe bond in this compound and it is analagous to $Co(CO)_8$. These compounds can be regarded as complexes of the $RS^-$ ion, in which the sulphur atoms share two electron pairs with the metal atoms. The $RS^-$ bridges found in these compounds seem stronger than halogen bridges. In compounds where $RS^-$ acts as a bridge between two metal atoms, it is tercovalent and pyramidal. A review of compounds containing sulphur ligands has been written (Livingstone, 1965).

If the $(MeZnSBu^t)_5$ molecule is regarded as being formed from $RS^-$ and $RZn^+$ ions then S1 is tercovalent and pyramidal as found in several compounds mentioned by Livingstone. However, S2, S3 and S4 are four-covalent with tetrahedral coordination, and S5 shares three electron pairs to bridge four zinc atoms and is five-coordinate.

Compounds in which $RS^-$ bridges three or four metal atoms are not well known. However, in $(C_5H_5FeS)_4$, sulphur bridges three iron atoms (C.J. Fritchie, Jr., C.T. Prewitt, R.A. Schunn and R.M. Sweet, 1966) and in the sulphides of cadmium and zinc the sulphur is surrounded by four metal atoms at tetrahedral corners. In $OSF_4$ (Bauer and Kimura, 1963) the sulphur atom is five-coordinate with a trigonal bipyramidal arrangement of atoms around it.

In the compounds $(MeZnSR)_n$, the value of n is found to depend on the nature of R. When R is t-butyl or i-propyl the molecular weights in benzene solution correspond to degrees of association of five and six respectively. If R is methyl, n-propyl or phenyl, the compounds are insoluble in benzene and believed to be polymeric. It would appear that the degree of association varies inversely with the bulkiness of the groups bonded to the α-carbon atom. There is no evidence to show that the steric interactions between organic groups in $(MeZnSBu^t)_5$, is great since only eight of the intramolecular contacts between the carbon atoms of different alkyl groups are less than 4.0Å. On steric grounds there seems to be no reason why an MeZn unit should not be accommodated at the eighth corner of the cube. Indeed it seems that the presence of the fifth $MeZnSBu^t$ unit near this location distorts the structure in such a way that the intramolecular contacts involving C1 and C2 are shorter than they would be in a cubane-type arrangement with Zn2 at the eighth corner.

The compounds $(MeZnOR)_4$ are tetrameric when R is Me,$Bu^t$,$Pr^i$ or Ph, and are probably isostructural with $(MeZnOMe)_4$. The mean Zn-O distance in $(MeZnOMe)_4$ is 2.09 Å (Shearer and Spencer, 1966a) which is smaller than the Zn-S distance in $(MeZnSBu^t)_5$. Thus the steric interaction between the organic groups bonded to zinc and oxygen will be more pronounced, leading to lower degrees of polymerisation.

The change from the cubane-type structure of $(MeZnOMe)_4$ to the pentameric arrangement found in $(MeZnSBu^t)_5$ is of interest. In the cubane-type of arrangement the bond angles in the central part of the molecule must be near 90° and it resembles part of an NaCl type lattice. The Zn-S and Zn-O bond lengths in $(MeZnOMe)_4$ and $(MeZnSBu^t)_5$ indicate a large ionic contribution to the bonding. The radius ratios of the central atoms in $(MeZnSBu^t)_5$, $(MeZnOMe)_4$, $(MeCdSBu^t)_4$ and $(MeCdOMe)_4$ are 0.40, 0.53, 0.53, and 0.69. The tetrameric cubane-type structure is found in $(MeZnOMe)_4$, and $(MeCdSBu^t)_4$ and $(MeCdOMe)_4$ probably also have this structure. The ratio of $Zn^{++}/S^{--}$, however is outside the range in which the NaCl type lattice is stable. Thus the deductions which can be made from a consideration of radius ratio are in agreement with the observed degrees of association.

$(MeZnSBu^t)_5$

Table 8.

Final Values of the Observed and Calculated Structure Factors.

| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
| 0 | 2 | 0 | --- | -20 |
| 0 | 4 | 0 | 360 | -418 |
| 0 | 6 | 0 | 11 | -14 |
| 0 | 8 | 0 | 84 | -96 |
| 0 | 10 | 0 | 107 | -103 |
| 0 | 12 | 0 | 123 | 117 |
| 0 | 14 | 0 | 189 | 190 |
| 0 | 16 | 0 | < 7 | -21 |
| 0 | 18 | 0 | 261 | -284 |
| 0 | 20 | 0 | 27 | 32 |
| 0 | 22 | 0 | 67 | 58 |
| 0 | 24 | 0 | 29 | -30 |
| 0 | 26 | 0 | 77 | 79 |
| 0 | 28 | 0 | 26 | -23 |
| 0 | 30 | 0 | 38 | -33 |
| 0 | 32 | 0 | 38 | 25 |
| 0 | 34 | 0 | 20 | 21 |
| 0 | 1 | 1 | 173 | 156 |
| 0 | 2 | 1 | 361 | 423 |
| 0 | 3 | 1 | 148 | -134 |
| 0 | 4 | 1 | 61 | 67 |
| 0 | 5 | 1 | < 6 | -7 |
| 0 | 6 | 1 | < 4 | -3 |
| 0 | 7 | 1 | 186 | -181 |
| 0 | 8 | 1 | 76 | -86 |
| 0 | 9 | 1 | 101 | -97 |
| 0 | 10 | 1 | 82 | -88 |
| 0 | 11 | 1 | 133 | 134 |
| 0 | 12 | 1 | 30 | 34 |
| 0 | 13 | 1 | 144 | 189 |
| 0 | 14 | 1 | 80 | -87 |
| 0 | 15 | 1 | 47 | -55 |
| 0 | 16 | 1 | 172 | 175 |
| 0 | 17 | 1 | 107 | -109 |
| 0 | 18 | 1 | 82 | 88 |
| 0 | 19 | 1 | 13 | 15 |
| 0 | 20 | 1 | 141 | -146 |
| 0 | 21 | 1 | 68 | 70 |
| 0 | 22 | 1 | < 9 | -6 |
| 0 | 23 | 1 | 40 | -41 |
| 0 | 24 | 1 | < 9 | -5 |
| 0 | 25 | 1 | < 10 | -1 |
| 0 | 26 | 1 | 28 | 27 |
| 0 | 27 | 1 | 28 | 28 |
| 0 | 28 | 1 | 21 | 21 |
| 0 | 29 | 1 | 48 | -53 |
| 0 | 30 | 1 | 19 | -14 |
| 0 | 31 | 1 | < 11 | -12 |
| 0 | 32 | 1 | < 11 | 8 |
| 0 | 33 | 1 | 22 | 21 |
| 0 | 34 | 1 | 32 | -32 |
| 0 | 35 | 1 | < 12 | 2 |
| 0 | 36 | 1 | < 12 | 8 |
| 0 | 37 | 1 | < 12 | -2 |
| 0 | 38 | 1 | 21 | 16 |
| 0 | 0 | 2 | 84 | 82 |
| 0 | 1 | 2 | 43 | -38 |
| 0 | 2 | 2 | 26 | -24 |
| 0 | 3 | 2 | 66 | -89 |
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| h | k | l | \|Fo\| | Fc |
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| 3 | 8 | 6 | 23 | -19 |
| 3 | 9 | 6 | 29 | -30 |
| 3 | 10 | 6 | 33 | -34 |
| 3 | 11 | 6 | < 9 | 9 |
| 3 | 12 | 6 | < 9 | 8 |
| 3 | 13 | 6 | 22 | 29 |
| 3 | 14 | 6 | 24 | 29 |
| 3 | 15 | 6 | < 9 | -11 |
| 3 | 16 | 6 | < 9 | -3 |
| 3 | 17 | 6 | < 9 | -19 |
| 3 | 18 | 6 | < 9 | -12 |
| 3 | 19 | 6 | < 9 | 0 |
| 3 | 20 | 6 | 21 | 26 |
| 3 | 21 | 6 | 13 | 10 |
| 3 | 22 | 6 | 16 | -19 |
| 3 | 23 | 6 | < 9 | 6 |
| 3 | 24 | 6 | 20 | -21 |
| 3 | 25 | 6 | < 9 | -3 |
| 3 | 26 | 6 | 18 | 19 |
| 3 | 1 | 7 | 17 | 16 |
| 3 | 2 | 7 | 38 | 32 |
| 3 | 3 | 7 | 31 | -33 |
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| 3 | 5 | 7 | < 9 | -4 |
| 3 | 6 | 7 | 18 | 16 |
| 3 | 7 | 7 | 52 | 50 |
| 3 | 8 | 7 | < 9 | -5 |
| 3 | 9 | 7 | < 9 | -8 |
| 3 | 10 | 7 | 19 | -13 |
| 3 | 11 | 7 | 44 | -46 |
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| 3 | 13 | 7 | < 9 | 4 |
| 3 | 14 | 7 | 22 | -9 |
| 3 | 0 | 8 | 45 | -49 |
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| 3 | 2 | 8 | < 9 | -4 |
| 3 | 3 | 8 | 15 | 8 |
| 3 | 4 | 8 | 15 | 21 |
| 3 | 5 | 8 | 12 | -18 |
| 3 | 1 | 9 | 35 | -9 |
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| 4 | 2 | -12 | < 8 | 4 |
| 4 | 3 | -12 | < 8 | 8 |
| 4 | 4 | -12 | < 8 | 21 |
| 4 | 5 | -12 | < 8 | 10 |
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| 4 | 10 | -12 | 19 | 22 |
| 4 | 11 | -12 | 11 | 11 |
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| 4 | 13 | -12 | 25 | 12 |
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| 4 | 1 | -11 | < 9 | -10 |
| 4 | 2 | -11 | 12 | -18 |
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| 4 | 4 | -11 | 12 | 15 |
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| 4 | 7 | -11 | 21 | -13 |
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| 4 | 10 | -11 | 21 | -38 |
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| 4 | 13 | -11 | < 9 | -14 |
| 4 | 14 | -11 | 17 | 8 |
| 4 | 15 | -11 | 17 | 19 |
| 4 | 0 | -10 | 17 | -14 |
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| 4 | 2 | -9 | < 9 | 10 |
| 4 | 3 | -9 | 29 | 31 |
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| 4 | 4 | -8 | < 8 | 1 |
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| 4 | 8 | -8 | < 8 | -4 |
| 4 | 9 | -8 | 23 | -23 |
| 4 | 10 | -8 | < 8 | -7 |
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| 4 | 14 | -8 | < 9 | 12 |
| 4 | 15 | -8 | 45 | 44 |
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| 4 | 5 | -7 | 99 | 83 |
| 4 | 6 | -7 | 64 | -54 |
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| 4 | 8 | -7 | 72 | 64 |
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| 4 | 13 | -7 | 129 | -109 |
| 4 | 14 | -7 | < 8 | -12 |
| 4 | 15 | -7 | 39 | 38 |
| 4 | 16 | -7 | 70 | 61 |
| 4 | 17 | -7 | 87 | 84 |
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| 4 | 22 | -7 | 15 | 12 |
| 4 | 23 | -7 | 18 | -19 |
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| 4 | 25 | -7 | < 9 | -7 |
| 4 | 26 | -7 | 22 | -21 |
| 4 | 27 | -7 | < 9 | -4 |
| 4 | 28 | -7 | 26 | 21 |
| 4 | 29 | -7 | < 9 | 16 |
| 4 | 30 | -7 | < 9 | 13 |
| 4 | 31 | -7 | 30 | 28 |
| 4 | 32 | -7 | 18 | -14 |
| 4 | 0 | -6 | 291 | -229 |
| 4 | 1 | -6 | 77 | 63 |
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| 4 | 3 | -6 | 123 | -90 |
| 4 | 4 | -6 | 198 | 152 |
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| 4 | 8 | -6 | 35 | -37 |
| 4 | 9 | -6 | 66 | 68 |
| 4 | 10 | -6 | 36 | 41 |
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| 4 | 12 | -6 | 17 | -23 |
| 4 | 13 | -6 | 38 | -38 |
| 4 | 14 | -6 | 92 | -83 |
| 4 | 15 | -6 | 43 | -48 |
| 4 | 16 | -6 | < 8 | 3 |
| 4 | 17 | -6 | 21 | 19 |
| 4 | 18 | -6 | 63 | 62 |
| 4 | 19 | -6 | 18 | -14 |
| 4 | 20 | -6 | 28 | 28 |
| 4 | 21 | -6 | 63 | 51 |
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| 4 | 25 | -6 | 58 | -64 |
| 4 | 26 | -6 | < 9 | -9 |
| 4 | 27 | -6 | 16 | 17 |
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| 4 | 2 | -5 | 98 | -83 |
| 4 | 3 | -5 | 52 | 47 |
| 4 | 4 | -5 | 202 | -166 |
| 4 | 5 | -5 | 130 | -113 |
| 4 | 6 | -5 | 72 | 64 |
| 4 | 7 | -5 | 41 | 43 |
| 4 | 8 | -5 | 162 | 108 |
| 4 | 9 | -5 | 21 | 26 |
| 4 | 10 | -5 | 18 | -19 |
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| 4 | 13 | -5 | 16 | 13 |
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| 4 | 15 | -5 | < 6 | -5 |
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| 4 | 17 | -5 | < 7 | -2 |
| 4 | 18 | -5 | 33 | -30 |
| 4 | 19 | -5 | < 8 | 1 |
| 4 | 20 | -5 | 28 | 33 |
| 4 | 21 | -5 | 32 | -35 |
| 4 | 22 | -5 | 74 | 65 |
| 4 | 23 | -5 | 39 | 42 |
| 4 | 24 | -5 | 41 | -40 |
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| 4 | 26 | -5 | 20 | -21 |
| 4 | 27 | -5 | 24 | -23 |
| 4 | 28 | -5 | 28 | 28 |
| 4 | 29 | -5 | 17 | 17 |
| 4 | 0 | -4 | < 5 | 0 |
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| 4 | 2 | -4 | 84 | 71 |
| 4 | 3 | -4 | 47 | 47 |
| 4 | 4 | -4 | 28 | -29 |
| 4 | 5 | -4 | 53 | -47 |
| 4 | 6 | -4 | 82 | -74 |
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| 4 | 8 | -4 | 76 | 57 |
| 4 | 9 | -4 | 117 | 115 |
| 4 | 10 | -4 | 18 | -26 |
| 4 | 11 | -4 | 56 | -53 |
| 4 | 12 | -4 | 33 | 41 |
| 4 | 13 | -4 | 66 | -68 |
| 4 | 14 | -4 | < 6 | 8 |
| 4 | 15 | -4 | < 6 | 3 |
| 4 | 16 | -4 | 19 | -24 |
| 4 | 17 | -4 | 21 | 21 |
| 4 | 18 | -4 | < 6 | 1 |
| 4 | 19 | -4 | 30 | 33 |
| 4 | 20 | -4 | 14 | -17 |
| 4 | 21 | -4 | < 7 | -3 |
| 4 | 22 | -4 | < 8 | -7 |
| 4 | 23 | -4 | 21 | -17 |
| 4 | 24 | -4 | 26 | 31 |
| 4 | 25 | -4 | < 8 | 0 |
| 4 | 26 | -4 | 21 | -18 |
| 4 | 1 | -3 | 121 | -88 |
| 4 | 2 | -3 | 107 | -89 |
| 4 | 3 | -3 | 20 | 21 |
| 4 | 4 | -3 | 11 | -13 |
| 4 | 5 | -3 | 39 | 48 |
| 4 | 6 | -3 | 25 | 26 |
| 4 | 7 | -3 | 6 | 5 |
| 4 | 8 | -3 | 32 | 38 |
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| 4 | 10 | -3 | 12 | 12 |
| 4 | 11 | -3 | 21 | 18 |
| 4 | 12 | -3 | 32 | -42 |
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| 4 | 17 | -3 | 40 | -4[illegible] |

| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
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| 4 | 19 | -3 | 66 | 55 |
| 4 | 20 | -3 | 86 | 84 |
| 4 | 21 | -3 | 81 | 49 |
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| 4 | 25 | -3 | < 8 | 1 |
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| 4 | 0 | -2 | --- | 209 |
| 4 | 1 | -2 | --- | -66 |
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| 4 | 5 | -2 | 77 | -61 |
| 4 | 6 | -2 | 23 | 28 |
| 4 | 7 | -2 | 114 | 91 |
| 4 | 8 | -2 | 107 | 79 |
| 4 | 9 | -2 | 106 | 90 |
| 4 | 10 | -2 | 46 | -36 |
| 4 | 11 | -2 | 41 | 38 |
| 4 | 12 | -2 | 36 | -43 |
| 4 | 13 | -2 | 26 | -26 |
| 4 | 14 | -2 | 69 | 63 |
| 4 | 15 | -2 | 71 | -77 |
| 4 | 16 | -2 | 13 | -12 |
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| 4 | 21 | -2 | 79 | 79 |
| 4 | 22 | -2 | < 7 | 9 |
| 4 | 23 | -2 | 30 | 28 |
| 4 | 24 | -2 | < 8 | 6 |
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| 4 | 2 | -1 | 174 | 123 |
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| 4 | 5 | -1 | 240 | -183 |
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| 4 | 7 | -1 | 43 | -40 |
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| 4 | 9 | -1 | < 5 | 3 |
| 4 | 10 | -1 | 136 | 120 |
| 4 | 11 | -1 | 27 | 27 |
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| 4 | 14 | -1 | 131 | -109 |
| 4 | 15 | -1 | 16 | -15 |
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| 4 | 17 | -1 | 140 | -131 |
| 4 | 18 | -1 | < 8 | -2 |
| 4 | 19 | -1 | < 8 | -13 |
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| 4 | 21 | -1 | < 7 | 7 |
| 4 | 22 | -1 | 30 | 31 |
| 4 | 23 | -1 | 18 | 21 |
| 4 | 24 | -1 | 13 | -8 |
| 4 | 25 | -1 | 22 | 25 |
| 4 | 26 | -1 | 14 | -13 |
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| 4 | 28 | -1 | < 8 | 7 |
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| 4 | 30 | -1 | < 9 | -1 |
| 4 | 31 | -1 | 23 | -27 |
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| 4 | 1 | 0 | 17 | -13 |
| 4 | 2 | 0 | 80 | 82 |
| 4 | 3 | 0 | 79 | 62 |
| 4 | 4 | 0 | 43 | -43 |
| 4 | 5 | 0 | 105 | 86 |
| 4 | 6 | 0 | 128 | -92 |
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| 4 | 9 | 0 | 46 | -47 |
| 4 | 10 | 0 | < 6 | 3 |
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| 4 | 12 | 0 | 176 | 184 |
| 4 | 13 | 0 | 11 | 14 |
| 4 | 14 | 0 | 18 | 23 |
| 4 | 15 | 0 | 94 | 94 |
| 4 | 16 | 0 | 164 | -153 |
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| 4 | 18 | 0 | < 7 | 3 |
| 4 | 19 | 0 | < 7 | -1 |
| 4 | 20 | 0 | 32 | 29 |
| 4 | 21 | 0 | 11 | 7 |
| 4 | 22 | 0 | < 8 | -1 |
| 4 | 23 | 0 | 16 | -17 |
| 4 | 24 | 0 | 26 | 30 |
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| 4 | 27 | 0 | < 9 | 3 |
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| 4 | 29 | 0 | 21 | 20 |
| 4 | 30 | 0 | 26 | -30 |
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| 4 | 32 | 0 | < 9 | 3 |
| 4 | 33 | 0 | < 9 | -14 |

| h | k | l | \|Fo\| | Fc |
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| 4 | 4 | 1 | 140 | 117 |
| 4 | 5 | 1 | 58 | -48 |
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| 4 | 11 | 1 | 39 | -48 |
| 4 | 12 | 1 | 56 | -63 |
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| 4 | 24 | 1 | 17 | 12 |
| 4 | 25 | 1 | 12 | 7 |
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| 4 | 10 | 2 | 46 | 86 |
| 4 | 11 | 2 | 34 | 23 |
| 4 | 12 | 2 | 42 | 46 |
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| 4 | 11 | 3 | 44 | 49 |
| 4 | 12 | 3 | 23 | 27 |
| 4 | 13 | 3 | 27 | 30 |
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| 4 | 15 | 3 | 18 | -16 |
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| 4 | 6 | 4 | 22 | 23 |
| 4 | 7 | 4 | 51 | -45 |
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| 4 | 12 | 4 | 21 | -24 |
| 4 | 13 | 4 | 21 | 28 |
| 4 | 14 | 4 | < 9 | 2 |
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| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
| 4 | 20 | 4 | < 9 | 10 |
| 4 | 21 | 4 | 26 | -24 |
| 4 | 22 | 4 | 19 | 21 |
| 4 | 23 | 4 | 20 | -21 |
| 4 | 24 | 4 | < 9 | -1 |
| 4 | 25 | 4 | 23 | 28 |
| 4 | 26 | 4 | 17 | -17 |
| 4 | 27 | 4 | 16 | 16 |
| 4 | 1 | 5 | 26 | -23 |
| 4 | 2 | 5 | < 9 | 7 |
| 4 | 3 | 5 | < 9 | -18 |
| 4 | 4 | 5 | 38 | 43 |
| 4 | 5 | 5 | 28 | 37 |
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| 4 | 9 | 5 | 18 | -16 |
| 4 | 10 | 5 | 23 | 21 |
| 4 | 11 | 5 | < 9 | -8 |
| 4 | 12 | 5 | < 9 | 1 |
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| 4 | 14 | 5 | 16 | 14 |
| 4 | 15 | 5 | 18 | -13 |
| 4 | 16 | 5 | < 9 | -3 |
| 4 | 17 | 5 | 9 | -3 |
| 4 | 18 | 5 | < 9 | -4 |
| 4 | 19 | 5 | 25 | 26 |
| 4 | 20 | 5 | < 9 | -16 |
| 4 | 21 | 5 | 20 | 19 |
| 4 | 22 | 5 | 16 | -16 |
| 4 | 23 | 5 | 16 | -18 |
| 4 | 24 | 5 | 20 | 27 |
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| 4 | 26 | 5 | 12 | 16 |
| 4 | 0 | 6 | 19 | 20 |
| 4 | 1 | 6 | 33 | -32 |
| 4 | 2 | 6 | 17 | -12 |
| 4 | 3 | 6 | < 9 | -9 |
| 4 | 4 | 6 | < 9 | 5 |
| 4 | 5 | 6 | 28 | 31 |
| 4 | 6 | 6 | 15 | 13 |
| 4 | 7 | 6 | < 9 | -5 |
| 4 | 8 | 6 | 19 | -17 |
| 4 | 9 | 6 | 17 | -21 |
| 4 | 1 | 7 | < 8 | 16 |
| 4 | 2 | 7 | < 8 | 22 |
| 4 | 3 | 7 | 19 | -23 |
| 4 | 0 | 8 | 19 | -28 |
| 5 | 0 | -12 | < 9 | -18 |
| 5 | 1 | -12 | < 9 | 1 |
| 5 | 2 | -12 | < 9 | 16 |
| 5 | 3 | -12 | < 9 | 7 |
| 5 | 4 | -12 | 13 | 15 |
| 5 | 5 | -12 | < 9 | -3 |
| 5 | 6 | -12 | 27 | -28 |
| 5 | 7 | -12 | < 9 | -9 |
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| 5 | 10 | -12 | 20 | 21 |
| 5 | 11 | -12 | < 9 | -4 |
| 5 | 12 | -12 | 18 | 16 |
| 5 | 1 | -11 | 13 | 19 |
| 5 | 2 | -11 | < 10 | 15 |
| 5 | 3 | -11 | < 10 | -11 |
| 5 | 4 | -11 | 31 | 27 |
| 5 | 5 | -11 | < 10 | -7 |
| 5 | 6 | -11 | < 10 | 3 |
| 5 | 7 | -11 | < 10 | -5 |
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| 5 | 11 | -11 | < 10 | 4 |
| 5 | 12 | -11 | < 10 | -2 |
| 5 | 13 | -11 | < 10 | 0 |
| 5 | 14 | -11 | 33 | 37 |
| 5 | 0 | -10 | < 8 | 1 |
| 5 | 1 | -10 | 43 | 51 |
| 5 | 2 | -10 | 16 | -18 |
| 5 | 3 | -10 | 44 | 46 |
| 5 | 4 | -10 | < 9 | -8 |
| 5 | 5 | -10 | 19 | -15 |
| 5 | 6 | -10 | 38 | 34 |
| 5 | 7 | -10 | 21 | -23 |
| 5 | 8 | -10 | 20 | 20 |
| 5 | 9 | -10 | < 9 | 0 |
| 5 | 10 | -10 | 18 | -21 |
| 5 | 11 | -10 | < 10 | -8 |
| 5 | 12 | -10 | 28 | -21 |
| 5 | 13 | -10 | < 10 | 1 |
| 5 | 14 | -10 | < 10 | 8 |
| 5 | 15 | -10 | 25 | 25 |
| 5 | 1 | -9 | 35 | -38 |
| 5 | 2 | -9 | 13 | 25 |
| 5 | 3 | -9 | 15 | 11 |
| 5 | 4 | -9 | 10 | -28 |
| 5 | 5 | -9 | 23 | 27 |
| 5 | 6 | -9 | 22 | -26 |
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| 5 | 8 | -9 | 22 | 23 |
| 5 | 9 | -9 | 24 | -29 |

| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
| 5 | 10 | -9 | 20 | 14 |
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| 5 | 12 | -9 | 20 | -25 |
| 5 | 13 | -9 | < 10 | 16 |
| 5 | 14 | -9 | 22 | 30 |
| 5 | 15 | -9 | 18 | -10 |
| 5 | 16 | -9 | < 10 | 11 |
| 5 | 17 | -9 | < 10 | 7 |
| 5 | 18 | -9 | 32 | -35 |
| 5 | 19 | -9 | 15 | 19 |
| 5 | 0 | -8 | 53 | 44 |
| 5 | 1 | -8 | < 7 | -7 |
| 5 | 2 | -8 | < 7 | 5 |
| 5 | 3 | -8 | 15 | 17 |
| 5 | 4 | -8 | 45 | -38 |
| 5 | 5 | -8 | < 7 | -4 |
| 5 | 6 | -8 | 21 | 23 |
| 5 | 7 | -8 | 15 | -17 |
| 5 | 8 | -8 | 12 | 12 |
| 5 | 9 | -8 | 15 | 20 |
| 5 | 10 | -8 | 39 | -38 |
| 5 | 11 | -8 | 48 | -48 |
| 5 | 12 | -8 | < 9 | -1 |
| 5 | 13 | -8 | < 10 | 16 |
| 5 | 14 | -8 | 24 | 23 |
| 5 | 15 | -8 | 56 | 52 |
| 5 | 16 | -8 | 17 | 15 |
| 5 | 17 | -8 | 34 | -35 |
| 5 | 18 | -8 | < 10 | -6 |
| 5 | 19 | -8 | < 10 | -6 |
| 5 | 20 | -8 | 18 | -16 |
| 5 | 21 | -8 | 18 | 18 |
| 5 | 1 | -7 | 24 | -20 |
| 5 | 2 | -7 | 182 | 146 |
| 5 | 3 | -7 | 68 | -62 |
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| 5 | 5 | -7 | 74 | 77 |
| 5 | 6 | -7 | 90 | -77 |
| 5 | 7 | -7 | 47 | 43 |
| 5 | 8 | -7 | 30 | 34 |
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| 5 | 11 | -7 | < 9 | -14 |
| 5 | 12 | -7 | 112 | -86 |
| 5 | 13 | -7 | 28 | -28 |
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| 5 | 15 | -7 | < 9 | 6 |
| 5 | 16 | -7 | 49 | 48 |
| 5 | 17 | -7 | 9 | 16 |
| 5 | 18 | -7 | < 9 | -8 |
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| 5 | 22 | -7 | < 10 | -7 |
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| 5 | 26 | -6 | 24 | 25 |
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| 5 | 18 | -3 | 15 | 22 |
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| 5 | 20 | -3 | 21 | 18 |
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| 5 | 24 | -3 | 13 | 14 |
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| 5 | 5 | -2 | < 6 | -8 |
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| 5 | 14 | -2 | 32 | 38 |
| 5 | 15 | -2 | 102 | -88 |
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| 5 | 18 | -2 | 54 | -48 |
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| 5 | 23 | -2 | < 9 | 10 |
| 5 | 24 | -2 | 24 | -23 |
| 5 | 25 | -2 | < 9 | -1 |
| 5 | 26 | -2 | 39 | 38 |
| 5 | 27 | -2 | 13 | 9 |

| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
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| h | k | l | \|Fo\| | Fc |
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| 5 | 21 | 2 | 33 | 43 |
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| 5 | 6 | 3 | 68 | 59 |
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| 5 | 12 | 3 | 17 | 18 |
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| 6 | 7 | -10 | 18 | -17 |
| 6 | 8 | -10 | 17 | 18 |
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| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
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| 6 | 17 | -5 | 40 | 35 |

| h | k | l | \|Fo\| | Fc |
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| 6 | 19 | -5 | 28 | 28 |
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| 6 | 21 | -5 | 29 | -23 |
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| 6 | 27 | -5 | < 11 | -3 |
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| 6 | 4 | -2 | 22 | -26 |
| 6 | 5 | -2 | < 7 | -4 |
| 6 | 6 | -2 | 18 | 15 |
| 6 | 7 | -2 | 77 | -72 |
| 6 | 8 | -2 | 51 | -45 |
| 6 | 9 | -2 | 18 | 34 |
| 6 | 10 | -2 | 30 | 29 |
| 6 | 11 | -2 | 50 | 43 |
| 6 | 12 | -2 | < 8 | 8 |
| 6 | 13 | -2 | 20 | 19 |
| 6 | 14 | -2 | 17 | 14 |
| 6 | 15 | -2 | 9 | -9 |
| 6 | 16 | -2 | 27 | 26 |
| 6 | 17 | -2 | 16 | -15 |
| 6 | 18 | -2 | 36 | -37 |
| 6 | 19 | -2 | < 10 | -1 |

| h | k | l | \|Fo\| | Fo |
|---|---|---|---|---|
| 6 | 20 | -2 | 10 | -14 |
| 6 | 21 | -2 | 24 | -20 |
| 6 | 22 | -2 | < 10 | 1 |
| 6 | 23 | -2 | < 10 | -12 |
| 6 | 24 | -2 | < 10 | 0 |
| 6 | 25 | -2 | 40 | 41 |
| 6 | 26 | -2 | 21 | 14 |
| 6 | 1 | -1 | < 9 | -4 |
| 6 | 2 | -1 | 13 | -15 |
| 6 | 3 | -1 | < 9 | -6 |
| 6 | 4 | -1 | 18 | -12 |
| 6 | 5 | -1 | 48 | 53 |
| 6 | 6 | -1 | < 9 | 5 |
| 6 | 7 | -1 | 38 | -38 |
| 6 | 8 | -1 | < 9 | -10 |
| 6 | 9 | -1 | 42 | -37 |
| 6 | 10 | -1 | < 9 | -3 |
| 6 | 11 | -1 | 23 | 21 |
| 6 | 12 | -1 | 13 | 14 |
| 6 | 13 | -1 | < 9 | 3 |
| 6 | 14 | -1 | < 9 | -2 |
| 6 | 15 | -1 | 31 | 28 |
| 6 | 16 | -1 | 14 | 13 |
| 6 | 17 | -1 | < 10 | -3 |
| 6 | 18 | -1 | 20 | -19 |
| 6 | 19 | -1 | 18 | -18 |
| 6 | 20 | -1 | < 10 | -7 |
| 6 | 21 | -1 | < 10 | 5 |
| 6 | 22 | -1 | 32 | 33 |
| 6 | 23 | -1 | 24 | -21 |
| 6 | 24 | -1 | < 11 | 4 |
| 6 | 25 | -1 | < 11 | 0 |
| 6 | 26 | -1 | < 11 | -14 |
| 6 | 27 | -1 | 19 | 19 |
| 6 | 0 | 0 | < 10 | -22 |
| 6 | 1 | 0 | 14 | 21 |
| 6 | 2 | 0 | 10 | 17 |
| 6 | 3 | 0 | 14 | -16 |
| 6 | 4 | 0 | < 10 | 2 |
| 6 | 5 | 0 | 17 | -17 |
| 6 | 6 | 0 | 26 | -26 |
| 6 | 7 | 0 | 14 | 20 |
| 6 | 8 | 0 | < 10 | 6 |
| 6 | 9 | 0 | 14 | 13 |
| 6 | 10 | 0 | 34 | 46 |
| 6 | 11 | 0 | < 10 | 1 |
| 6 | 12 | 0 | 18 | -17 |
| 6 | 13 | 0 | 20 | -19 |
| 6 | 14 | 0 | 34 | -34 |
| 6 | 15 | 0 | < 10 | -1 |
| 6 | 16 | 0 | 31 | 34 |
| 6 | 17 | 0 | < 11 | 13 |
| 6 | 18 | 0 | < 11 | -3 |
| 6 | 19 | 0 | 22 | -22 |
| 6 | 20 | 0 | 59 | -58 |
| 6 | 21 | 0 | 19 | 16 |
| 6 | 22 | 0 | < 11 | 13 |
| 6 | 23 | 0 | < 11 | 13 |
| 6 | 24 | 0 | 40 | 41 |
| 6 | 1 | 1 | 28 | -29 |
| 6 | 2 | 1 | 18 | 17 |
| 6 | 3 | 1 | 44 | 44 |
| 6 | 4 | 1 | 28 | 24 |
| 6 | 5 | 1 | 11 | -6 |
| 6 | 6 | 1 | 26 | -28 |
| 6 | 7 | 1 | 35 | -40 |
| 6 | 8 | 1 | 18 | -22 |
| 6 | 9 | 1 | < 11 | 6 |
| 6 | 10 | 1 | 15 | 17 |
| 6 | 11 | 1 | 46 | 53 |
| 6 | 12 | 1 | < 11 | 13 |
| 6 | 13 | 1 | < 11 | 2 |
| 6 | 14 | 1 | < 11 | 3 |
| 6 | 15 | 1 | < 11 | -17 |
| 6 | 16 | 1 | < 11 | -4 |
| 6 | 17 | 1 | < 11 | 4 |
| 6 | 18 | 1 | < 11 | 10 |
| 6 | 19 | 1 | 27 | -34 |
| 6 | 20 | 1 | < 11 | -4 |
| 6 | 21 | 1 | < 11 | -13 |
| 6 | 22 | 1 | 23 | -25 |
| 6 | 23 | 1 | 28 | 29 |
| 6 | 0 | 2 | 19 | -20 |
| 6 | 1 | 2 | < 11 | -2 |
| 6 | 2 | 2 | < 11 | -1 |
| 6 | 3 | 2 | 24 | 37 |
| 6 | 4 | 2 | 24 | 26 |
| 6 | 5 | 2 | 27 | 29 |
| 6 | 6 | 2 | < 11 | 2 |
| 6 | 7 | 2 | 11 | -10 |
| 6 | 8 | 2 | 31 | -30 |
| 6 | 9 | 2 | 29 | -36 |
| 6 | 10 | 2 | < 11 | 21 |
| 6 | 11 | 2 | 29 | -35 |
| 6 | 12 | 2 | < 11 | 2 |
| 6 | 13 | 2 | 25 | 24 |
| 6 | 14 | 2 | < 11 | -8 |
| 6 | 15 | 2 | 28 | 37 |
| 6 | 16 | 2 | 26 | 26 |
| 6 | 17 | 2 | 16 | 2 |

| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
| 6 | 1 | 3 | 11 | -14 |
| 6 | 2 | 3 | 11 | 18 |
| 6 | 3 | 3 | 15 | -15 |
| 6 | 4 | 3 | 15 | 19 |
| 6 | 5 | 3 | 27 | 33 |
| 6 | 6 | 3 | 11 | 21 |
| 6 | 7 | 3 | < 11 | 9 |
| 6 | 8 | 3 | 22 | -21 |
| 6 | 9 | 3 | < 11 | 2 |
| 6 | 10 | 3 | 19 | -19 |
| 6 | 11 | 3 | < 11 | -5 |
| 6 | 12 | 3 | < 11 | 0 |
| 6 | 13 | 3 | 41 | -44 |
| 6 | 14 | 3 | < 11 | -9 |
| 6 | 15 | 3 | 20 | 21 |
| 6 | 16 | 3 | < 11 | 10 |
| 6 | 17 | 3 | 23 | 27 |
| 6 | 18 | 3 | 19 | 0 |

## THE CRYSTAL STRUCTURE OF METHYLZINC ISOPROPYLSULPHIDE

### Introduction

The compound is produced when equimolar amounts of dimethylzinc and isopropylthiol react at -70°C. (Coates and Ridley, 1965).

$$Me_2Zn + Pr^iSH \longrightarrow MeZnSPr^i + CH_4$$

The molecular weight of this compound as determined cryoscopically in benzene solution corresponds to degrees of association of 6.05, 6.05, 6.20 and 5.90 at concentrations of 1.66, 1.97, 2.03 and 2.49 wt.%. Methylcadmium isopropylsulphide (Coates and Lauder, 1966) was found to have molecular weights in benzene solution which corresponded to degrees of association of 6.10, 6.09, 5.98 and 6.03 at concentrations of 3.75, 4.33, 5.11 and 6.24 wt.%. Both compounds are thus believed to be hexameric in benzene solution. Coates and Lauder (1966) proposed a structure for the cadmium compound in which the hexameric molecule is made up of six-membered rings with alternate cadmium and sulphur atoms. Two such rings are held together by bonds between the cadmium atoms of one ring and the sulphur atoms of the other. All of the cadmium and sulphur atoms are four-coordinate.

This structure analysis was started after the structure of $(MeZnSBu^t)_5$ had been determined. It was thought that a comparison of the structures of $(MeZnSBu^t)_5$ and $(MeZnSPr^i)_6$

would help to explain the different degrees of association and structures of these compounds.

## Experimental.

### Preliminary X-ray Study

Crystals suitable for X-ray analysis were obtained by recrystallisation from hexane and were sealed in pyrex-glass capillary tubes in the dry nitrogen atmosphere of a glove-box. The crystals were of irregular shape, with poorly developed faces but were approximately rectangular prisms.

The unit cell and space group were found from precession photographs of the hk0, hk1, h01 and h11 reciprocal lattice nets. The errors in unit cell length given by a statistical treatment of the measurements from the photographs were

0.001 Å in $\underline{a}$ , and 0.0008 Å in $\underline{c}$. Independent measurements of $\underline{a}$ on two films gave values which differed by 0.1%. When allowance is made for systematic errors, the uncertainty in the unit cell lengths is probably of the order of 0.25%.

The crystals are tetragonal.

$\underline{a} = 13.61$ Å, $\underline{c} = 15.16$ Å.

Conditions limiting observed reflections,

hol, $h + 1 = 2n$,

hko, $h + k = 2n$.

Space group $P4_2/nnm$. $D_{4h}^{12}$ No. 134.

$$D_m = 1.49 \text{ g.cm.}^{-3}$$

If there are 16 units of $CH_3Zn\,S\,C_3H_7$ per unit cell the density would be 1.473 g.cm$^{-3}$. $F(000) = 1280e$.

Linear absorption coefficient Cu K$\alpha$. radiation = 67.3.cm.$^{-1}$.

## Data Collection and Correction

It was noted that the reflections given by crystals became weaker and more diffuse upon exposure to x-rays. Intensity data were recorded photographically using the equi-inclination Weissenberg technique and Cu.K$\alpha$ radiation. After each reciprocal lattice net had been photographed, an exposure of part of the okl net was made in order to check that there had been no marked deterioration of the crystal. Three crystals were used in collecting the intensity data. They were of irregular shape but their cross-sections were approximately rectangular. The dimensions of the cross-sections of the crystals used are given below.

| | |
|---|---|
| okl, 1kl, | 0.43 x 0.46 mm$^2$. |
| 2kl, | 0.12 x 0.14 mm$^2$. |
| 3kl, 4kl, 5kl, 6kl. | 0.36 x 0.40 mm$^2$. |

The intensities of reflections were measured visually by comparison with a graduated scale. They were corrected for Lorentz and polarisation factors and also, in the case of reflections on the upper layers, for spot length (Phillips, 1956). Corrections for absorption were applied by assuming that the crystals had a rectangular cross-section and summing over the cross-sectional area.

Crystals were mounted about a and equivalent reflections appeared on different nets. The data were put on a common scale by a least squares treatment of these reflections.

## Structure Determination

The corrected intensities were multiplied by a weighting function w, where w $= \exp\left(\frac{12 \sin^2\theta.}{\lambda^2}\right)$ This function was not allowed to take a value greater than 100.

The Patterson function was computed using the weighted intensities as coefficients. From an examination of the distribution of peaks in the Patterson function it seems probable that atoms lie at special positions. Several possible sets of positions for the zinc atoms have been found. While these arrangements satisfied many of the features of the Patterson function, none of them was fully consistent with it. Structure factors were calculated on these models, the lowest value of the residual obtained being 0.68.

An electron density map was calculated using the structure factors which gave a residual of 0.68. No useful deductions have been made from this electron density map.

The observed structure factors are shown in Table 1.

Methylzinc Isopropylsulphide.

Table 1.

Observed Structure Factors.

(Arbitrary units).

| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
| 0 | 0 | 2 | 90 | |
| 0 | 0 | 4 | 175 | |
| 0 | 0 | 6 | 189 | |
| 0 | 0 | 8 | 419 | |
| 0 | 0 | 10 | 84 | |
| 0 | 0 | 12 | 121 | |
| 0 | 0 | 14 | 68 | |
| 0 | 0 | 16 | 34 | |
| 0 | 0 | 18 | 8 | |
| 0 | 1 | 1 | 489 | |
| 0 | 1 | 3 | 61 | |
| 0 | 1 | 5 | 211 | |
| 0 | 1 | 7 | 352 | |
| 0 | 1 | 9 | 63 | |
| 0 | 1 | 11 | 22 | |
| 0 | 1 | 13 | 26 | |
| 0 | 1 | 15 | 47 | |
| 0 | 1 | 17 | < 7 | |
| 0 | 1 | 19 | 8 | |
| 0 | 2 | 0 | 213 | |
| 0 | 2 | 2 | 200 | |
| 0 | 2 | 4 | 22 | |
| 0 | 2 | 6 | 21 | |
| 0 | 2 | 8 | 63 | |
| 0 | 2 | 10 | 67 | |
| 0 | 2 | 12 | 42 | |
| 0 | 2 | 14 | 14 | |
| 0 | 2 | 16 | < 8 | |
| 0 | 3 | 1 | 35 | |
| 0 | 3 | 3 | 89 | |
| 0 | 3 | 5 | 21 | |
| 0 | 3 | 7 | 23 | |
| 0 | 3 | 9 | 52 | |
| 0 | 3 | 11 | 15 | |
| 0 | 3 | 13 | 11 | |
| 0 | 3 | 15 | < 8 | |
| 0 | 4 | 0 | 83 | |
| 0 | 4 | 2 | 84 | |
| 0 | 4 | 4 | 24 | |
| 0 | 4 | 6 | 10 | |
| 0 | 4 | 8 | < 7 | |
| 0 | 4 | 10 | < 8 | |
| 0 | 4 | 12 | 9 | |
| 0 | 5 | 1 | 45 | |
| 0 | 5 | 3 | 44 | |
| 0 | 5 | 5 | < 7 | |
| 0 | 5 | 7 | 30 | |
| 0 | 5 | 9 | 29 | |
| 0 | 5 | 11 | < 9 | |
| 0 | 5 | 13 | < 10 | |
| 0 | 5 | 15 | < 8 | |
| 0 | 5 | 17 | 16 | |
| 0 | 6 | 0 | 203 | |
| 0 | 6 | 2 | 70 | |
| 0 | 6 | 4 | 29 | |
| 0 | 6 | 6 | 28 | |
| 0 | 6 | 8 | 26 | |
| 0 | 6 | 10 | 36 | |
| 0 | 6 | 12 | 17 | |
| 0 | 6 | 14 | < 8 | |
| 0 | 6 | 16 | 19 | |
| 0 | 7 | 1 | 74 | |
| 0 | 7 | 3 | 181 | |
| 0 | 7 | 5 | 172 | |
| 0 | 7 | 7 | 13 | |
| 0 | 7 | 9 | 96 | |
| 0 | 7 | 11 | 33 | |
| 0 | 7 | 13 | 26 | |
| 0 | 8 | 0 | 46 | |
| 0 | 8 | 2 | 289 | |
| 0 | 8 | 4 | 39 | |
| 0 | 8 | 6 | 101 | |
| 0 | 8 | 8 | 41 | |
| 0 | 8 | 10 | 122 | |
| 0 | 8 | 12 | 30 | |
| 0 | 9 | 1 | 86 | |
| 0 | 9 | 3 | 108 | |
| 0 | 9 | 5 | 88 | |
| 0 | 9 | 7 | < 10 | |
| 0 | 9 | 9 | 74 | |
| 0 | 9 | 11 | 14 | |
| 0 | 10 | 0 | 34 | |
| 0 | 10 | 2 | 22 | |
| 0 | 10 | 4 | 13 | |
| 0 | 10 | 6 | 12 | |
| 0 | 10 | 8 | 10 | |
| 0 | 10 | 10 | 10 | |
| 0 | 11 | 1 | < 9 | |
| 0 | 11 | 3 | 20 | |
| 0 | 11 | 5 | 15 | |
| 0 | 11 | 7 | < 9 | |
| 0 | 11 | 9 | 9 | |
| 0 | 12 | 0 | 44 | |
| 0 | 12 | 2 | 19 | |
| 0 | 12 | 4 | 9 | |
| 0 | 12 | 6 | < 9 | |
| 0 | 12 | 8 | 20 | |
| 0 | 12 | 10 | 8 | |
| 0 | 13 | 1 | 17 | |
| 0 | 13 | 3 | < 9 | |

| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
| 0 | 13 | 5 | < 9 | |
| 0 | 13 | 7 | 16 | |
| 0 | 14 | 0 | < 8 | |
| 0 | 14 | 2 | 13 | |
| 0 | 15 | 1 | 14 | |
| 0 | 16 | 0 | 21 | |
| 0 | 16 | 2 | 8 | |
| 1 | 1 | 0 | --- | |
| 1 | 1 | 1 | 80 | |
| 1 | 1 | 2 | 240 | |
| 1 | 1 | 3 | 248 | |
| 1 | 1 | 4 | 134 | |
| 1 | 1 | 5 | 137 | |
| 1 | 1 | 6 | < 11 | |
| 1 | 1 | 7 | 22 | |
| 1 | 1 | 8 | 173 | |
| 1 | 1 | 9 | 42 | |
| 1 | 1 | 10 | 48 | |
| 1 | 1 | 11 | 39 | |
| 1 | 1 | 12 | 70 | |
| 1 | 1 | 13 | 23 | |
| 1 | 1 | 14 | 29 | |
| 1 | 1 | 15 | < 14 | |
| 1 | 1 | 16 | 12 | |
| 1 | 2 | 1 | 84 | |
| 1 | 2 | 2 | 187 | |
| 1 | 2 | 3 | 191 | |
| 1 | 2 | 4 | 190 | |
| 1 | 2 | 5 | 226 | |
| 1 | 2 | 6 | 117 | |
| 1 | 2 | 7 | 46 | |
| 1 | 2 | 8 | 13 | |
| 1 | 2 | 9 | 70 | |
| 1 | 2 | 10 | 21 | |
| 1 | 2 | 11 | < 13 | |
| 1 | 2 | 12 | 47 | |
| 1 | 2 | 13 | 20 | |
| 1 | 2 | 14 | 23 | |
| 1 | 3 | 0 | 113 | |
| 1 | 3 | 1 | 71 | |
| 1 | 3 | 2 | 40 | |
| 1 | 3 | 3 | 126 | |
| 1 | 3 | 4 | 11 | |
| 1 | 3 | 5 | 124 | |
| 1 | 3 | 6 | 38 | |
| 1 | 3 | 7 | 31 | |
| 1 | 3 | 8 | 76 | |
| 1 | 3 | 9 | 21 | |
| 1 | 3 | 10 | 39 | |
| 1 | 3 | 11 | 25 | |
| 1 | 3 | 12 | 11 | |
| 1 | 3 | 13 | 29 | |
| 1 | 3 | 14 | < 8 | |
| 1 | 3 | 15 | < 7 | |
| 1 | 3 | 16 | 13 | |
| 1 | 3 | 17 | 6 | |
| 1 | 4 | 1 | 46 | |
| 1 | 4 | 2 | 110 | |
| 1 | 4 | 3 | 88 | |
| 1 | 4 | 4 | 114 | |
| 1 | 4 | 5 | 78 | |
| 1 | 4 | 6 | 68 | |
| 1 | 4 | 7 | 44 | |
| 1 | 4 | 8 | 15 | |
| 1 | 4 | 9 | 24 | |
| 1 | 4 | 10 | 11 | |
| 1 | 4 | 11 | < 8 | |
| 1 | 4 | 12 | 31 | |
| 1 | 4 | 13 | 16 | |
| 1 | 4 | 14 | 11 | |
| 1 | 5 | 0 | 139 | |
| 1 | 5 | 1 | 48 | |
| 1 | 5 | 2 | 79 | |
| 1 | 5 | 3 | 76 | |
| 1 | 5 | 4 | 14 | |
| 1 | 5 | 5 | 98 | |
| 1 | 5 | 6 | 45 | |
| 1 | 5 | 7 | 41 | |
| 1 | 5 | 8 | 60 | |
| 1 | 5 | 9 | < 10 | |
| 1 | 5 | 10 | 29 | |
| 1 | 5 | 11 | 14 | |
| 1 | 5 | 12 | < 10 | |
| 1 | 5 | 13 | 20 | |
| 1 | 5 | 14 | < 10 | |
| 1 | 5 | 15 | < 9 | |
| 1 | 5 | 16 | 11 | |
| 1 | 6 | 1 | 87 | |
| 1 | 6 | 2 | 71 | |
| 1 | 6 | 3 | 35 | |
| 1 | 6 | 4 | 111 | |
| 1 | 6 | 5 | 87 | |
| 1 | 6 | 6 | 68 | |
| 1 | 6 | 7 | 78 | |
| 1 | 6 | 8 | < 14 | |
| 1 | 6 | 9 | < 11 | |
| 1 | 6 | 10 | < 11 | |
| 1 | 6 | 11 | 11 | |
| 1 | 6 | 12 | 28 | |
| 1 | 7 | 0 | 66 | |

| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
| 1 | 7 | 1 | 35 | |
| 1 | 7 | 2 | 177 | |
| 1 | 7 | 3 | 69 | |
| 1 | 7 | 4 | 34 | |
| 1 | 7 | 5 | 67 | |
| 1 | 7 | 6 | 60 | |
| 1 | 7 | 7 | < 15 | |
| 1 | 7 | 8 | 15 | |
| 1 | 7 | 9 | < 16 | |
| 1 | 7 | 10 | 74 | |
| 1 | 7 | 11 | 22 | |
| 1 | 7 | 12 | 24 | |
| 1 | 7 | 13 | 25 | |
| 1 | 8 | 1 | 80 | |
| 1 | 8 | 2 | 13 | |
| 1 | 8 | 3 | 167 | |
| 1 | 8 | 4 | < 14 | |
| 1 | 8 | 5 | 182 | |
| 1 | 8 | 6 | < 15 | |
| 1 | 8 | 7 | < 15 | |
| 1 | 8 | 8 | < 16 | |
| 1 | 8 | 9 | 92 | |
| 1 | 8 | 10 | < 17 | |
| 1 | 8 | 11 | 27 | |
| 1 | 8 | 12 | < 15 | |
| 1 | 8 | 13 | 17 | |
| 1 | 9 | 0 | 80 | |
| 1 | 9 | 1 | 13 | |
| 1 | 9 | 2 | 111 | |
| 1 | 9 | 3 | 62 | |
| 1 | 9 | 4 | 47 | |
| 1 | 9 | 5 | 35 | |
| 1 | 9 | 6 | 29 | |
| 1 | 9 | 7 | 16 | |
| 1 | 9 | 8 | < 17 | |
| 1 | 9 | 9 | < 17 | |
| 1 | 9 | 10 | 80 | |
| 1 | 9 | 11 | 25 | |
| 1 | 9 | 12 | 18 | |
| 1 | 10 | 1 | 32 | |
| 1 | 10 | 2 | 29 | |
| 1 | 10 | 3 | < 15 | |
| 1 | 10 | 4 | 42 | |
| 1 | 10 | 5 | < 15 | |
| 1 | 10 | 6 | 24 | |
| 1 | 10 | 7 | 33 | |
| 1 | 11 | 0 | < 15 | |
| 1 | 11 | 1 | < 15 | |
| 1 | 11 | 2 | 47 | |
| 1 | 11 | 3 | 21 | |
| 1 | 11 | 4 | < 15 | |
| 1 | 11 | 5 | < 15 | |
| 1 | 11 | 6 | 21 | |
| 1 | 12 | 1 | 15 | |
| 1 | 12 | 2 | < 15 | |
| 1 | 12 | 3 | 21 | |
| 1 | 13 | 0 | 34 | |
| 1 | 13 | 1 | < 15 | |
| 1 | 13 | 2 | < 15 | |
| 1 | 13 | 3 | 20 | |
| 1 | 15 | 0 | 17 | |
| 2 | 2 | 0 | --- | |
| 2 | 2 | 1 | 46 | |
| 2 | 2 | 2 | 41 | |
| 2 | 2 | 3 | 49 | |
| 2 | 2 | 4 | 18 | |
| 2 | 2 | 5 | 185 | |
| 2 | 2 | 6 | 94 | |
| 2 | 2 | 7 | 90 | |
| 2 | 2 | 8 | 122 | |
| 2 | 2 | 9 | < 12 | |
| 2 | 2 | 10 | 40 | |
| 2 | 2 | 11 | 27 | |
| 2 | 2 | 12 | 14 | |
| 2 | 2 | 13 | 34 | |
| 2 | 3 | 1 | 123 | |
| 2 | 3 | 2 | 8 | |
| 2 | 3 | 3 | 20 | |
| 2 | 3 | 4 | 27 | |
| 2 | 3 | 5 | 63 | |
| 2 | 3 | 6 | 36 | |
| 2 | 3 | 7 | 136 | |
| 2 | 3 | 8 | < 7 | |
| 2 | 3 | 9 | 66 | |
| 2 | 3 | 10 | 18 | |
| 2 | 3 | 11 | 17 | |
| 2 | 3 | 12 | 18 | |
| 2 | 3 | 13 | < 14 | |
| 2 | 3 | 14 | 14 | |
| 2 | 3 | 15 | 21 | |
| 2 | 4 | 0 | 187 | |
| 2 | 4 | 1 | 15 | |
| 2 | 4 | 2 | 139 | |
| 2 | 4 | 3 | 8 | |
| 2 | 4 | 4 | 223 | |
| 2 | 4 | 5 | 14 | |
| 2 | 4 | 6 | 28 | |
| 2 | 4 | 7 | 11 | |
| 2 | 4 | 8 | 49 | |
| 2 | 4 | 9 | 11 | |

| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
| 2 | 4 | 10 | 52 | |
| 2 | 4 | 11 | 14 | |
| 2 | 4 | 12 | 33 | |
| 2 | 5 | 1 | 123 | |
| 2 | 5 | 2 | 32 | |
| 2 | 5 | 3 | 135 | |
| 2 | 5 | 4 | 44 | |
| 2 | 5 | 5 | 135 | |
| 2 | 5 | 6 | 47 | |
| 2 | 5 | 7 | 21 | |
| 2 | 5 | 8 | 17 | |
| 2 | 5 | 9 | 78 | |
| 2 | 5 | 10 | < 14 | |
| 2 | 5 | 11 | 20 | |
| 2 | 5 | 12 | 20 | |
| 2 | 6 | 0 | 117 | |
| 2 | 6 | 1 | 58 | |
| 2 | 6 | 2 | 92 | |
| 2 | 6 | 3 | 80 | |
| 2 | 6 | 4 | < 10 | |
| 2 | 6 | 5 | 110 | |
| 2 | 6 | 6 | 53 | |
| 2 | 6 | 7 | 31 | |
| 2 | 6 | 8 | 63 | |
| 2 | 6 | 9 | 11 | |
| 2 | 6 | 10 | 37 | |
| 2 | 6 | 11 | 25 | |
| 2 | 6 | 12 | 16 | |
| 2 | 6 | 13 | 23 | |
| 2 | 7 | 1 | 51 | |
| 2 | 7 | 2 | 71 | |
| 2 | 7 | 3 | 39 | |
| 2 | 7 | 4 | 81 | |
| 2 | 7 | 5 | 68 | |
| 2 | 7 | 6 | 60 | |
| 2 | 7 | 7 | 63 | |
| 2 | 8 | 0 | 86 | |
| 2 | 8 | 1 | < 12 | |
| 2 | 8 | 2 | 64 | |
| 2 | 8 | 3 | 14 | |
| 2 | 8 | 4 | 24 | |
| 2 | 8 | 5 | 18 | |
| 2 | 8 | 6 | < 13 | |
| 2 | 8 | 7 | < 14 | |
| 2 | 8 | 8 | 28 | |
| 2 | 8 | 9 | < 14 | |
| 2 | 8 | 10 | 31 | |
| 2 | 8 | 11 | < 14 | |
| 2 | 8 | 12 | 29 | |
| 2 | 9 | 1 | 24 | |
| 2 | 9 | 2 | 46 | |
| 2 | 9 | 3 | 20 | |
| 2 | 9 | 4 | 84 | |
| 2 | 9 | 5 | 25 | |
| 2 | 9 | 6 | 14 | |
| 2 | 9 | 7 | 35 | |
| 2 | 9 | 8 | < 14 | |
| 2 | 9 | 9 | < 14 | |
| 2 | 9 | 10 | 33 | |
| 2 | 10 | 0 | 88 | |
| 2 | 10 | 1 | 18 | |
| 2 | 10 | 2 | 28 | |
| 2 | 10 | 3 | 48 | |
| 2 | 10 | 4 | < 14 | |
| 2 | 10 | 5 | 24 | |
| 2 | 10 | 6 | 28 | |
| 2 | 10 | 7 | 20 | |
| 2 | 10 | 8 | 22 | |
| 2 | 10 | 9 | < 14 | |
| 2 | 10 | 10 | 20 | |
| 2 | 11 | 1 | 26 | |
| 2 | 11 | 2 | < 14 | |
| 2 | 11 | 3 | 37 | |
| 2 | 11 | 4 | < 14 | |
| 2 | 11 | 5 | 37 | |
| 2 | 12 | 0 | 31 | |
| 2 | 12 | 1 | < 14 | |
| 2 | 12 | 2 | 20 | |
| 2 | 13 | 1 | 20 | |
| 2 | 14 | 0 | 14 | |
| 3 | 3 | 0 | 482 | |
| 3 | 3 | 1 | 26 | |
| 3 | 3 | 2 | 84 | |
| 3 | 3 | 3 | 133 | |
| 3 | 3 | 4 | 78 | |
| 3 | 3 | 5 | 79 | |
| 3 | 3 | 6 | < 7 | |
| 3 | 3 | 7 | 60 | |
| 3 | 3 | 8 | 168 | |
| 3 | 3 | 9 | 22 | |
| 3 | 3 | 10 | 80 | |
| 3 | 3 | 11 | 24 | |
| 3 | 3 | 12 | 63 | |
| 3 | 3 | 13 | 11 | |
| 3 | 3 | 14 | 26 | |
| 3 | 3 | 15 | 14 | |
| 3 | 3 | 16 | 14 | |
| 3 | 3 | 17 | 8 | |
| 3 | 4 | 1 | 114 | |
| 3 | 4 | 2 | 95 | |

| h | k | l | \|Fo\| | Fc |
|---|---|---|---|---|
| 3 | 4 | 3 | 158 | |
| 3 | 4 | 4 | 172 | |
| 3 | 4 | 5 | 179 | |
| 3 | 4 | 6 | 90 | |
| 3 | 4 | 7 | 162 | |
| 3 | 4 | 8 | 34 | |
| 3 | 4 | 9 | 39 | |
| 3 | 4 | 10 | 47 | |
| 3 | 4 | 11 | < 8 | |
| 3 | 4 | 12 | 44 | |
| 3 | 4 | 13 | 28 | |
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| 3 | 4 | 17 | 8 | |
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| 3 | 5 | 1 | 16 | |
| 3 | 5 | 2 | 245 | |
| 3 | 5 | 3 | 72 | |
| 3 | 5 | 4 | 54 | |
| 3 | 5 | 5 | 60 | |
| 3 | 5 | 6 | 56 | |
| 3 | 5 | 7 | 28 | |
| 3 | 5 | 8 | 15 | |
| 3 | 5 | 9 | 12 | |
| 3 | 5 | 10 | 81 | |
| 3 | 5 | 11 | 21 | |
| 3 | 5 | 12 | 29 | |
| 3 | 5 | 13 | 17 | |
| 3 | 5 | 14 | 9 | |
| 3 | 6 | 1 | 74 | |
| 3 | 6 | 2 | 42 | |
| 3 | 6 | 3 | 77 | |
| 3 | 6 | 4 | 45 | |
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| 3 | 6 | 8 | < 9 | |
| 3 | 6 | 9 | 58 | |
| 3 | 6 | 10 | < 9 | |
| 3 | 6 | 11 | 16 | |
| 3 | 6 | 12 | < 8 | |
| 3 | 6 | 13 | 10 | |
| 3 | 6 | 14 | 7 | |
| 3 | 7 | 0 | 61 | |
| 3 | 7 | 1 | 28 | |
| 3 | 7 | 2 | 53 | |
| 3 | 7 | 3 | 69 | |
| 3 | 7 | 4 | < 5 | |
| 3 | 7 | 5 | 71 | |
| 3 | 7 | 6 | 16 | |
| 3 | 7 | 7 | 14 | |
| 3 | 7 | 8 | 41 | |
| 3 | 7 | 9 | < 9 | |
| 3 | 7 | 10 | 16 | |
| 3 | 7 | 11 | 17 | |
| 3 | 7 | 12 | < 8 | |
| 3 | 7 | 13 | 16 | |
| 3 | 7 | 14 | 10 | |
| 3 | 8 | 1 | 78 | |
| 3 | 8 | 2 | < 7 | |
| 3 | 8 | 3 | 18 | |
| 3 | 8 | 4 | 11 | |
| 3 | 8 | 5 | 15 | |
| 3 | 8 | 6 | < 9 | |
| 3 | 8 | 7 | 60 | |
| 3 | 9 | 0 | 59 | |
| 3 | 9 | 1 | 8 | |
| 3 | 9 | 2 | < 8 | |
| 3 | 9 | 3 | 60 | |
| 3 | 9 | 4 | 10 | |
| 3 | 9 | 5 | 22 | |
| 3 | 9 | 6 | 29 | |
| 3 | 9 | 7 | 24 | |
| 3 | 9 | 8 | 41 | |
| 3 | 9 | 9 | < 9 | |
| 3 | 9 | 10 | < 9 | |
| 3 | 9 | 11 | 17 | |
| 3 | 10 | 1 | 30 | |
| 3 | 10 | 2 | < 8 | |
| 3 | 10 | 3 | 36 | |
| 3 | 10 | 4 | 17 | |
| 3 | 10 | 5 | 33 | |
| 3 | 10 | 6 | < 9 | |
| 3 | 10 | 7 | 17 | |
| 3 | 10 | 8 | 14 | |
| 3 | 10 | 9 | 32 | |
| 3 | 10 | 10 | 13 | |
| 3 | 10 | 11 | 12 | |
| 3 | 11 | 0 | < 8 | |
| 3 | 11 | 1 | 26 | |
| 3 | 11 | 2 | 67 | |
| 3 | 11 | 3 | 30 | |
| 3 | 11 | 4 | 12 | |
| 3 | 11 | 5 | 38 | |
| 3 | 11 | 6 | 24 | |
| 3 | 11 | 7 | < 8 | |
| 3 | 11 | 8 | < 8 | |
| 3 | 11 | 9 | < 8 | |
| 3 | 11 | 10 | 35 | |
| 3 | 12 | 1 | < 8 | |
| 3 | 12 | 2 | 32 | |
| 3 | 12 | 3 | 35 | |
| 3 | 12 | 4 | −31 | |
| 3 | 12 | 5 | 28 | |
| 3 | 12 | 6 | 30 | |
| 3 | 12 | 7 | 20 | |
| 3 | 12 | 8 | < 8 | |
| 3 | 12 | 9 | 14 | |
| 3 | 13 | 0 | 33 | |
| 3 | 13 | 1 | < 8 | |
| 3 | 13 | 2 | 14 | |
| 3 | 14 | 1 | 14 | |
| 3 | 15 | 0 | < 6 | |
| 3 | 15 | 1 | < 6 | |
| 3 | 15 | 2 | 15 | |
| 3 | 15 | 3 | 11 | |
| 3 | 16 | 1 | < 5 | |
| 3 | 16 | 2 | < 5 | |
| 3 | 16 | 3 | 16 | |
| 3 | 16 | 4 | 11 | |
| 4 | 4 | 0 | 365 | |
| 4 | 4 | 1 | 119 | |
| 4 | 4 | 2 | 183 | |
| 4 | 4 | 3 | 198 | |
| 4 | 4 | 4 | 57 | |
| 4 | 4 | 5 | 191 | |
| 4 | 4 | 6 | 16 | |
| 4 | 4 | 7 | 70 | |
| 4 | 4 | 8 | 93 | |
| 4 | 4 | 9 | 35 | |
| 4 | 4 | 10 | 83 | |
| 4 | 4 | 11 | 73 | |
| 4 | 4 | 12 | 80 | |
| 4 | 4 | 13 | 50 | |
| 4 | 4 | 14 | 15 | |
| 4 | 4 | 15 | 20 | |
| 4 | 5 | 1 | 47 | |
| 4 | 5 | 2 | 134 | |
| 4 | 5 | 3 | 181 | |
| 4 | 5 | 4 | 186 | |
| 4 | 5 | 5 | 168 | |
| 4 | 5 | 6 | 75 | |
| 4 | 5 | 7 | 107 | |
| 4 | 5 | 8 | < 9 | |
| 4 | 5 | 9 | 41 | |
| 4 | 5 | 10 | 22 | |
| 4 | 5 | 11 | 9 | |
| 4 | 5 | 12 | 39 | |
| 4 | 5 | 13 | 19 | |
| 4 | 5 | 14 | 18 | |
| 4 | 5 | 15 | 14 | |
| 4 | 5 | 16 | < 6 | |
| 4 | 5 | 17 | 10 | |
| 4 | 6 | 0 | 114 | |
| 4 | 6 | 1 | 13 | |
| 4 | 6 | 2 | 49 | |
| 4 | 6 | 3 | 7 | |
| 4 | 6 | 4 | 33 | |
| 4 | 6 | 5 | 17 | |
| 4 | 6 | 6 | < 8 | |
| 4 | 6 | 7 | < 8 | |
| 4 | 6 | 8 | 31 | |
| 4 | 6 | 9 | < 9 | |
| 4 | 6 | 10 | 39 | |
| 4 | 6 | 11 | < 9 | |
| 4 | 6 | 12 | 16 | |
| 4 | 6 | 13 | < 8 | |
| 4 | 6 | 14 | < 7 | |
| 4 | 6 | 15 | 10 | |
| 4 | 7 | 1 | 10 | |
| 4 | 7 | 2 | 16 | |
| 4 | 7 | 3 | < 8 | |
| 4 | 7 | 4 | 29 | |
| 4 | 7 | 5 | < 8 | |
| 4 | 7 | 6 | 22 | |
| 4 | 7 | 7 | < 9 | |
| 4 | 7 | 8 | 14 | |
| 4 | 8 | 0 | 34 | |
| 4 | 9 | 1 | 8 | |
| 4 | 9 | 2 | 20 | |
| 4 | 9 | 3 | < 8 | |
| 4 | 9 | 4 | 32 | |
| 4 | 9 | 5 | < 9 | |
| 4 | 9 | 6 | 25 | |
| 4 | 10 | 0 | 36 | |
| 4 | 10 | 1 | < 8 | |
| 4 | 10 | 2 | 17 | |
| 4 | 10 | 3 | < 8 | |
| 4 | 10 | 4 | < 8 | |
| 4 | 10 | 5 | 12 | |
| 4 | 10 | 6 | < 9 | |
| 4 | 10 | 7 | < 9 | |
| 4 | 10 | 8 | 15 | |
| 4 | 11 | 1 | 8 | |
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| 4 | 11 | 3 | 33 | |
| 4 | 11 | 4 | 45 | |
| 4 | 11 | 5 | 38 | |
| 4 | 11 | 6 | 33 | |
| 4 | 11 | 7 | 19 | |
| 4 | 11 | 8 | < 8 | |
| 4 | 11 | 9 | 10 | |
| 4 | 11 | 10 | 10 | |
| 4 | 11 | 11 | < 7 | |
| 4 | 11 | 12 | 13 | |
| 4 | 12 | 0 | 30 | |
| 4 | 12 | 1 | 29 | |
| 4 | 12 | 2 | 41 | |
| 4 | 12 | 3 | 43 | |
| 4 | 12 | 4 | 10 | |
| 4 | 12 | 5 | 45 | |
| 4 | 12 | 6 | < 8 | |
| 4 | 12 | 7 | 14 | |
| 4 | 12 | 8 | < 8 | |
| 4 | 12 | 9 | < 7 | |
| 4 | 12 | 10 | 24 | |
| 4 | 12 | 11 | 18 | |
| 4 | 12 | 12 | 11 | |
| 4 | 12 | 13 | 9 | |
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| 4 | 13 | 2 | 14 | |
| 4 | 13 | 3 | 14 | |
| 4 | 13 | 4 | 20 | |
| 4 | 13 | 5 | 15 | |
| 4 | 13 | 6 | 15 | |
| 4 | 13 | 7 | < 7 | |
| 4 | 13 | 8 | 15 | |
| 5 | 5 | 0 | 180 | |
| 5 | 5 | 1 | 16 | |
| 5 | 5 | 2 | 83 | |
| 5 | 5 | 3 | 88 | |
| 5 | 5 | 4 | 87 | |
| 5 | 5 | 5 | 79 | |
| 5 | 5 | 6 | < 10 | |
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| 5 | 5 | 8 | 81 | |
| 5 | 5 | 9 | < 11 | |
| 5 | 5 | 10 | 22 | |
| 5 | 5 | 11 | < 10 | |
| 5 | 5 | 12 | 31 | |
| 5 | 5 | 13 | 19 | |
| 5 | 5 | 14 | 13 | |
| 5 | 6 | 1 | 107 | |
| 5 | 6 | 2 | 24 | |
| 5 | 6 | 3 | 10 | |
| 5 | 6 | 4 | 44 | |
| 5 | 6 | 5 | 19 | |
| 5 | 6 | 6 | < 10 | |
| 5 | 6 | 7 | 74 | |
| 5 | 6 | 8 | 11 | |
| 5 | 6 | 9 | 16 | |
| 5 | 6 | 10 | 27 | |
| 5 | 6 | 11 | 15 | |
| 5 | 6 | 12 | 10 | |
| 5 | 7 | 0 | 55 | |
| 5 | 7 | 1 | < 9 | |
| 5 | 7 | 2 | 47 | |
| 5 | 7 | 3 | 56 | |
| 5 | 7 | 4 | < 10 | |
| 5 | 7 | 5 | .27 | |
| 5 | 7 | 6 | 35 | |
| 5 | 7 | 7 | 31 | |
| 5 | 7 | 8 | 19 | |
| 5 | 7 | 9 | < 12 | |
| 5 | 7 | 10 | 25 | |
| 5 | 7 | 11 | 18 | |
| 5 | 7 | 12 | 14 | |
| 5 | 8 | 1 | 21 | |
| 5 | 8 | 2 | < 10 | |
| 5 | 8 | 3 | 38 | |
| 5 | 8 | 4 | < 10 | |
| 5 | 8 | 5 | 36 | |
| 5 | 8 | 6 | < 11 | |
| 5 | 8 | 7 | < 11 | |
| 5 | 8 | 8 | 16 | |
| 5 | 8 | 9 | 20 | |
| 5 | 8 | 10 | 16 | |
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| 5 | 9 | 2 | 45 | |
| 5 | 9 | 3 | 34 | |
| 5 | 9 | 4 | < 10 | |
| 5 | 9 | 5 | 36 | |
| 5 | 9 | 6 | 25 | |
| 5 | 9 | 7 | 16 | |
| 5 | 9 | 8 | 23 | |
| 5 | 9 | 9 | < 12 | |
| 5 | 9 | 10 | < 11 | |
| 5 | 9 | 11 | 14 | |
| 5 | 10 | 1 | 50 | |
| 5 | 10 | 2 | 21 | |
| 5 | 10 | 3 | < 10 | |
| 5 | 10 | 4 | 28 | |
| 5 | 10 | 5 | < 11 | |
| 5 | 10 | 6 | < 11 | |
| 5 | 10 | 7 | 40 | |
| 5 | 10 | 8 | 11 | |
| 5 | 10 | 9 | 21 | |
| 5 | 10 | 10 | 20 | |
| 5 | 11 | 0 | 55 | |
| 5 | 11 | 1 | < 10 | |
| 5 | 11 | 2 | < 10 | |
| 5 | 11 | 3 | < 10 | |
| 5 | 11 | 4 | 10 | |
| 5 | 11 | 5 | 10 | |
| 5 | 11 | 6 | < 10 | |
| 5 | 11 | 7 | < 10 | |
| 5 | 11 | 8 | 24 | |
| 5 | 12 | 1 | < 10 | |
| 5 | 12 | 2 | 25 | |
| 5 | 12 | 3 | 20 | |
| 5 | 12 | 4 | 20 | |
| 5 | 12 | 5 | 22 | |
| 5 | 12 | 6 | 14 | |
| 5 | 12 | 7 | 13 | |
| 5 | 13 | 0 | < 10 | |
| 5 | 13 | 1 | < 10 | |
| 5 | 13 | 2 | 28 | |
| 5 | 15 | 0 | 10 | |
| 6 | 6 | 0 | 49 | |
| 6 | 6 | 1 | 24 | |
| 6 | 6 | 2 | 31 | |
| 6 | 6 | 3 | 138 | |
| 6 | 6 | 4 | < 11 | |
| 6 | 6 | 5 | 37 | |
| 6 | 6 | 6 | 41 | |
| 6 | 6 | 7 | 24 | |
| 6 | 6 | 8 | 30 | |
| 6 | 6 | 9 | 16 | |
| 6 | 6 | 10 | 21 | |
| 6 | 6 | 11 | 20 | |
| 6 | 7 | 1 | < 10 | |
| 6 | 7 | 2 | 37 | |
| 6 | 7 | 3 | 41 | |
| 6 | 7 | 4 | 62 | |
| 6 | 7 | 5 | 36 | |
| 6 | 7 | 6 | 21 | |
| 6 | 7 | 7 | 18 | |
| 6 | 7 | 8 | 15 | |
| 6 | 7 | 9 | 16 | |
| 6 | 7 | 10 | 15 | |
| 6 | 8 | 0 | 57 | |
| 6 | 8 | 1 | 11 | |
| 6 | 8 | 2 | 33 | |
| 6 | 8 | 3 | < 12 | |
| 6 | 8 | 4 | < 12 | |
| 6 | 8 | 5 | < 12 | |
| 6 | 8 | 6 | < 13 | |
| 6 | 8 | 7 | < 13 | |
| 6 | 8 | 8 | 18 | |
| 6 | 9 | 1 | 11 | |
| 6 | 9 | 2 | 25 | |
| 6 | 9 | 3 | 23 | |
| 6 | 9 | 4 | 23 | |
| 6 | 9 | 5 | 21 | |
| 6 | 9 | 6 | 21 | |
| 6 | 9 | 7 | < 12 | |
| 6 | 9 | 8 | < 12 | |
| 6 | 9 | 9 | 12 | |
| 6 | 10 | 0 | 44 | |
| 6 | 10 | 1 | 16 | |
| 6 | 10 | 2 | 28 | |
| 6 | 10 | 3 | 31 | |
| 6 | 10 | 4 | < 12 | |
| 6 | 10 | 5 | 33 | |
| 6 | 10 | 6 | 19 | |
| 6 | 10 | 7 | < 12 | |
| 6 | 10 | 8 | 16 | |
| 6 | 10 | 9 | < 11 | |
| 6 | 10 | 10 | 14 | |
| 6 | 11 | 1 | 28 | |
| 6 | 11 | 2 | 11 | |
| 6 | 12 | 0 | 16 | |
| 6 | 12 | 1 | < 11 | |
| 6 | 12 | 2 | 15 | |

## COMPUTER PROGRAMMES

I have written the following programmes in Algol for an Elliott 803B computer. Some optimization procedures and machine orders were used. Matrix operations were programmed using procedures, in some cases modified, from the Elliott Algol Matrix Package.

### Data Correction Programme

This programme corrects intensity data collected by the equi-inclination Weissenberg Technique for anisotropic absorption, Lorentz and polarization factors and also, in the case of upper net photographs, for spot length. The method used for evaluating the absorption correction is essentially that of Busing and Levy (1957).

The transmission coefficient, A, is calculated by evaluating the expression

$$A = \int\left(\frac{1}{V}\right)\exp\left[-\mu(r_{\alpha} + r_{\beta})\right] dV \qquad 1$$

where V is the volume of the crystal, $\mu$ the absorption coefficient, and $r_{\alpha}$ and $r_{\beta}$ are the pathlengths of the incident and diffracted rays respectively. The crystal must have plane surfaces and no re-entrant angles. The surfaces are represented by inequalities of the type

$$a_s x + b_s y + c_s z - d_s \geq 0 \qquad 2$$

A point inside the crystal will satisfy all of the inequalities, whereas a point outside will not. The reciprocal lattice and the crystal faces are referred to the same set of orthogonal axes, X, Y and Z, $a^*$ must lie along X, $b^*$ must lie in the XY plane, c must lie along Z, the rotation axis. The crystal axes may have to be renamed to satisfy these conditions.

The integration in equation 1 is evaluated by doing the summation.

$$\sum_{i=1}^{m_x} \sum_{j=1}^{m_y} \sum_{k=1}^{m_z} (b-a) \times \left[d(x_i) - c(x_i)\right] \times \left[f(x_i, y_j) - e(x_i, y_j)\right] \times R_i R_j R_k \exp\left[-\mu(r_\alpha + r_\beta)\right] \tag{3}$$

The subscripts $x_i$, $y_j$ and $z_k$ refer to a point in the crystal to and from which pathlengths of incident and diffracted rays are calculated.

$$x_i = a + (b-a) u_i \tag{4}$$

$$y_j = c(x_i) + \left[d(x_i) - c(x_i)\right] u_j \tag{5}$$

$$z_k = e(x_i, y_j) + \left[f(x_i, y_j) - e(x_i, y_j)\right] u_k \tag{6}$$

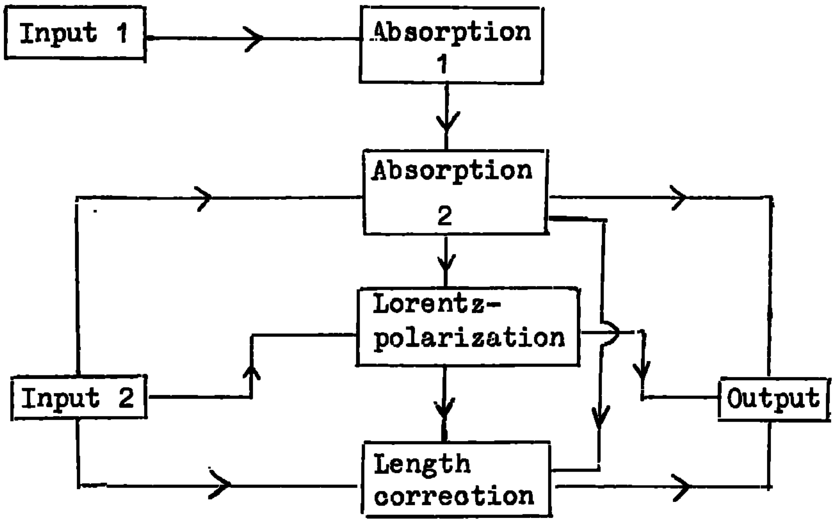
In equation 3;$m_x$, $m_y$ and $m_z$ are the numbers of elements into which the crystal is divided in the x, y and z directions respectively. The quantities a,b,c,d,e and f are limits and $R_i$, $R_j$, $R_k$, $u_i$,$u_j$ and $u_k$ are constants necessary for carrying out the summation.

The programme first takes all combinations of three of the inequalities and solves them for x so giving a and b, the minimum and maximum values of x respectively. Using equation 4 the values of $x_i$ are then calculated. For each value of $x_i$ the inequalities are taken in pairs and $c(x_i)$ and $d(x_i)$, the minimum and maximum values of y are found and hence using equation 5, the set of $y_j$. Similarly, the set of $z_k$ is found for each $(x_i, y_j)$.

The pathlengths are evaluated using the expressions

$$t_{\alpha s} = \frac{d_s - a_s x_i - b_s y_j - c_s z_k}{a_s \gamma_{\alpha x} + b_s \gamma_{\alpha y} + c_s \gamma_{\alpha z}} \quad , \quad t_{\beta s} = \frac{d_s - a_s x_i - b_s y_j - c_s z_k}{a_s \gamma_{\beta x} + b_s \gamma_{\beta y} + c_s \gamma_{\beta z}} \; .$$

The subscript s refers to a particular surface, $\gamma_\alpha$ and $\gamma_\beta$ are the direction cosines of the incident and diffracted rays respectively from the point $(x_i, y_j, x_k)$ to the surface s. Lengths of incident and diffracted beams are calculated from a point to each face. The shortest values of each are the correct ones. Direction cosines are calculated using the method of Wells (1960).



Flow diagram of data correction programme.

'Input 1' is only necessary if an absorption correction is to be applied and it reads the inequalities and also the constants $K_i$ and $u_i$.

'Absorption'1' is performed once for each crystal and it determines the limits of the summation and the coordinates of the points at which the transmission coefficient is to be calculated.

The numerators of equations 7 and 8, the weights (b-a)(d-c)(f-e) $R_iR_jR_k$ and the sum of the weights, which is equal to the volume, are also calculated and stored.

'Input 2' reads unit cell constants, scaling constant, linear absorption coefficient, reciprocal lattice constants and modifiers which relate Miller indices to the reference axes, describe the format of the intensity data and the corrections which are to be applied. The intensity data are provided in table form, the Miller indices of individual intensities are not required. Unobserved intensities are punched with a negative sign (e.g. $<3$ is punched as $-3$).

'Absorption 2' is done once for each reflection. The direction cosines of the incident and diffracted rays are calculated and used to evaluate expressions 7 and 8. With the numerators and weights calculated in 'Absorption1', expression 3 is evaluated and the summation carried out.

Lorentz and polarization correction and length correction (Phillips, 1956) are applied if specified and the corrected structure factor is multiplied by the specified scaling constant. The corrected structure factor is then punched out together with its' Miller indices. Unobserved structure factors are punched on a separate tape. The output is suitable for use with the Glasgow Fourier Summation and Structure Factor Least Squares programmes.

## Programme for determining film to film scaling constants

A number of methods have been published for determining the relative scaling constants for X-ray intensity data measured on several intersecting photographs. Kraut's method (1958) is only applicable when all of the intersecting reciprocal nets are obtained. Dickerson's method (1959) is applicable where some of the intersections are not obtained. In Dickerson's method the n scale factors are found from n equations of form.

$$\sum_{j=1}^{n} a_{ij} k_j = 0 \qquad i = 1 \text{ to } n$$

where

$$a_{ii} = \sum (F_{ij}^2)^2$$

$$a_{ij} = F_{ij}^2 \; F_{ji}^2 = a_{ji}$$

and $F_{ij}$ is the sum of the structure factors of all reflections on the i th film which are also observed on the jth film. In the resulting system of n equations in n unknowns the equations are not linearly independent. One equation is made redundant and one scale factor is set abitrarily at unity.

Rollett and Sparkes (1960) propose using coefficients of form

$$a_{ij} = - \sum_h W_h \, (F_{hi}^2) \, (F_{hj}^2)$$

$$a_{ii} = \sum W_h \, (F_{hi}^2)^2$$

Where h refers to a particular reflection and $W_h$ is a weight given to that reflection. They point out that the method of Dickerson was formally incorrect, the scale factors which result depending upon the choice of which scale factor is to be set at unity. They proposed the introduction of the condition that the sums of the squares of the scale factors should equal unity and that the scale factors should be determined by inverse iteration of the normal equations, the scale factors being normalised after each inversion.

The programme devised here follows the method of Rollett and Sparkes and will find scale factors for data collected about two or three axes. It is in two parts. The first forms coefficients of the type described above. The second part finds the scale factors by inverse iteration. In the work described the matrix elements were formed according to Dickerson's formulae.

After this programme had been written Hamilton, Rollett and Sparkes (1965) described shortcomings in the methods mentioned above and suggested another in which the observational equations are not linear with respect to the scale factors. In the cases described in this thesis in which the above programme has been used the numbers of intersections have been large and under these circumstances the errors in scale factors resulting from using the formally incorrect method would be expected to be small.

## Appendix 1.

Elliott Algol version of

Data Correction Programme.

```
DATA CORRECTION PROGRAMME;
begin
comment matrix procedures;
procedure mxaux(a,b,c,d,e);
value d,e; Boolean d,e; array  a,b,c;
begin integer aa,ab,ac,sa;
aa:=address(a);ab:=address(b);
ac:=address(c);sa:=size(a);
if sa ≠ size(b) or sa ≠ size(c) then
begin print punch(3),££1?mxaux error?;
stop
end;
sa:=sa+aa-1;
for aa:=aa step 1 until sa do
begin location[aa]:=
if d then (if e then location[ab] else -location[ab])
else if e then location[ab]+location[ac]
else location[ab]-location[ac];
elliott(2,2,ab,0,2,2,ac)
end
end;
procedure mxcopy(a,b);
array a,b;
mxaux(a,b,b,true,true);
procedure mxquot(b,a,c,bb);
value a;array a,b,c;integer bb;
begin integer aa,ab,d,e,f,p,q,r,ra,rb,sa,x,xx,y,yy,
xstart,xstop;
real pivot,g;
switch ss:= 1;
mxcopy(b,c);
d:=aa:=address(a);ra:=range(a,1);
f:=ab:=address(b);rb:=range(b,2);
sa:=aa+size(a)-1;e:=ab-aa;
for p:=1 step 1 until ra do
begin pivot:=0;
x:=d;
for r:=p step 1 until ra do
begin if abs(location[x]) > abs(pivot) then
begin pivot:=location[x];
q:=r; y:=x
end;
x:=x+ra
end;
```

```
if pivot =0 then
begin
checks(£singular?);
bb:=1;
go to 1;
end;
xstop:=d+ra-p; x:=d+1;
if p ≠ q then
begin yy:=(q-1)*rb+ab;
for r :=d step 1 until xstop do
begin g:=location[r];
location[r]:=location[y]/pivot;
location[y]:=g; y:=y+1
end;
xstop:=f+rb-1;
for r:=f step 1 until xstop do
begin g:=location[r];
location[r]:=location[yy]/pivot;
location[yy]:=g;
yy:=yy+1
end
end
else begin for r:=d step 1 until xstop do
location[r]:=location[r]/pivot;
xstop :=f+rb-1;
for r:=f step 1 until xstop do
location[r]:=location[r]/pivot
end;
y:=d:=x+ra; xstart:=d-1;
xx:=f; yy:=f:=f+rb;
for r:=xstart step ra until sa do
begin g:=location[r];
for q:=p+1 step 1 until ra do
begin location[y]:=location[y]-g*location[x];
elliott(2,2,x,0,2,2,y)
end;
for q:=1 step 1 until rb do
begin location[yy]:=location[yy]-g*location[xx];
elliott(2,2,xx,0,2,2,yy)
end;
x:=x-ra+p; xx:=xx-rb;
y:=y+p
end
end;
xstart:=aa+ra-1;
xstop:=aa+1;x:=sa-ra;
```

```
for p:=xstart step (-1) until xstop do
begin yy:=ab;
for q:=p step ra until x do
begin g:=location[q];
for r:=1 step 1 until rb do
begin location[yy]:=location[yy]-g*location[xx];
elliott(2,2,yy,0,2,2,xx)
end;
xx:=xx-rb
end;
x:=x-ra; xx:=xx-rb
end;
l:
end;
procedure readvr(vector,n);
integer n;
array vector;
begin
  integer na;
  for na :=1 step 1 until n do
  read vector [na]
end;
precompile;
ABSORPTION CORRECTION;
begin
comment ABSORPTION ONE;
  integer na,nb,nc,nd,ne,nf,ng,rowmxa,p2,
          columa,xin,yin,zin,mx,my,mz,nn,rowa;
  real a,b,v,coefzb,vectzb;
  array coefxb[1:3,1:3],
        vectxb,vectxc[1:3,1:1],
        coefyb[1:2,1:2],
        vectyc,vectyd,vectyb[1:2,1:1],
        title[1:30];
  switch cc:=lb1;
  p2:=3;  elliott(7,0,0,0,2,3,p2);
  if p2=1 then go to lb1;
  nn:=1;  instring(title,nn);
  read rowmxa,mx,my,mz;
lb1:
begin
  integer bb;
  array coeffa[1:rowmxa,1:4],
        coefya[1:rowmxa,1:2],
        coefza,inside,vectza[1:rowmxa],
        vectya[1:rowmxa,1:1],
        xis,uisx,c,d,rx[1:mx],
          uisy,ry[1:my],
          rz,uisz[1:mz],
          e,f,yjs[1:mx,1:my],
          zks,weight[1:mx,1:my,1:mz],
          rn[1:mx,1:my,1:mz,1:rowmxa];
   switch cd:=lb2;
   if p2 =1 or p2=2  then go to lb2;
   for rowa :=1 step 1 until rowmxa do
   for columa :=1 step 1 until 4 do
   read coeffa[rowa,columa];
   readvr(uisx,mx);
   readvr(uisy,my);
   readvr(uisz,mz);
   readvr(rx,mx);
   readvr(ry,my);
   readvr(rz,mz);
   punch(2);
   print ££l4?absorption correction  £l2??;
   nn:=1;  outstring(title,nn);  print££l2??;
   advance(1);  if not buffer(1,£*?) then
   begin
     print £ quadrature data error ?;  wait;  restart
   end;
   for na:=1 step 1 until rowmxa do inside[na]:=
   (coeffa[na,4]+(0.000001*coeffa[na,4]));
   a:=b:=0;
   checks(£start of xis?);
   for na :=1 step 1 until (rowmxa-2) do
   for nb :=(na+1) step 1 until (rowmxa-1) do
   for nc :=(nb+1) step 1 until rowmxa do
 begin
 comment determines a and b and xis;
   for columa :=1,2,3 do
   begin
     coefxb[1,columa]:=coeffa[na,columa];
     coefxb[2,columa]:=coeffa[nb,columa];
     coefxb[3,columa]:=coeffa[nc,columa]
   end;
   vectxb[1,1]:=coeffa[na,4];
   vectxb[2,1]:=coeffa[nb,4];
   vectxb[3,1]:=coeffa[nc,4];
   bb:=0;
   mxquot(vectxc,coefxb,vectxb,bb);
   if bb=0 then
 begin
   xin :=0;
   for nd:=1 step 1 until rowmxa do
   if not (  coeffa[nd,1]*vectxc[1,1]+ coeffa[nd,2]*vectxc[2,1]
   + coeffa[nd,3]*vectxc[3,1] ≥ inside[nd] ) then xin:=xin+1;
```

```
  if xin=0 then
  begin
    if vectxc[1,1] < a then a:=vectxc[1,1]
    else if vectxc[1,1] > b then b:=vectxc[1,1]
  end
end
end;
  for na :=1 step 1 until mx do
  xis[na]:=checkr(a+(b-a)*uisx[na]);
  checks(£end of xis?);
  checks(£start of yjs?);
  for na := 1 step 1 until mx do
  begin
    d[na]:=0;
    c[na]:=0
  end;
  for na :=1 step 1 until mx do
begin
comment determines c and d and yj for each xi ;
  for nb :=1 step 1 until rowmxa do
  begin
    coefya[nb,1]:=coeffa[nb,2];
    coefya[nb,2]:=coeffa[nb,3];
    vectya[nb,1]:=coeffa[nb,4]-coeffa[nb,1]*xis[na]
  end;
  for nb :=1 step 1 until (rowmxa-1) do
  for nc :=(nb+1) step 1 until rowmxa do
begin
  for columa := 1,2 do
  begin
    coefyb[1,columa]:=coefya[nb,columa];
    coefyb[2,columa]:=coefya[nc,columa];
  end;
  vectyb[1,1]:=vectya[nb,1];
  vectyb[2,1]:=vectya[nc,1];
  bb:=0;
  mxquot(vectyc,coefyb,vectyb,bb);
  if bb=0 then
begin
  yin :=0;
  for nd :=1 step 1 until rowmxa do
  if not( coeffa[nd,1]*xis[na]+coeffa[nd,2]*vectyc[1,1]
  +coeffa[nd,3]*vectyc[2,1] ≥ inside[nd]) then yin:=yin+1;
```

```
     if yin=0 then
    begin
      if vectyc[1,1] > d[na] then d[na]:= vectyc[1,1]
      else if vectyc[1,1] < c[na] then c[na]:=vectyc[1,1]
    end
  end
  end
  end;
    for na := 1 step 1 until mx do
    for nb := 1 step 1 until my do
    yjs[na,nb]:= checkr(c[na]+(d[na]-c[na])*uisy[nb]);
    checks(£end of yjs?);
    checks(£start of zks?);for na :=1 step 1 until mx do
    for nb :=1 step 1 until my do
    begin
      e[na,nb]:=0;
      f[na,nb]:=0
    end;
    for na :=1 step 1 until mx do
    for nb :=1 step 1 until my do
  begin
  comment determines e and f and zks for each xi and yj;
    for rowa :=1 step 1 until rowmxa do
    begin
      coefza[rowa]:= coeffa[rowa,3];
      vectza[rowa]:= coeffa[rowa,4]-coeffa[rowa,1]*xis[na]
                     -coeffa[rowa,2]*yjs[na,nb]
    end;
    for nc :=1 step 1 until rowmxa do
  begin
    if vectza[nc] ≠ 0 and coefza[nc]  ≠ 0 then
  begin
    vectzb:= vectza[nc]/coefza[nc];
    zin := 0;
    for nd :=1 step 1 until rowmxa do
    if not( coeffa[nd,1]*xis[na]+coeffa[nd,2]*yjs[na,nb]
    +coeffa[nd,3]*vectzb ≥ inside[nd]) then zin:=zin+1;
    if zin =0 then
    begin
      if vectzb > f[na,nb] then f[na,nb]:= vectzb
      else if vectzb < e[na,nb] then e[na,nb]:=vectzb
    end
  end
  end
  end;
    v:=0;
    for na :=1 step 1 until mx do
    for nb :=1 step 1 until my do
    for nc :=1 step 1 until mz do
```

```
   zks[na,nb,nc]:=  checkr(
  e[na,nb]+(f[na,nb]-e[na,nb])*uisz[nc]);
  checks(£end of zks?);
  checks(£start of weights and numerators?);
  for na :=1 step 1 until mx do
  for nb :=1 step 1 until my do
  for nc :=1 step 1 until mz do
  begin
    weight[na,nb,nc]:= checkr(rx[na]*ry[nb]*rz[nc]*(b-a)
    *(d[na]-c[na])*(f[na,nb]-e[na,nb]));
    v := v + weight[na,nb,nc];
  for nd :=1 step 1 until rowmxa do
  rn[na,nb,nc,nd]:= checkr(-coeffa[nd,4]-
  coeffa[nd,1]*xis[na]-coeffa[nd,2]*yjs[na,nb]
  -coeffa[nd,3]*zks[na,nb,nc]);
  end;
  checks(£end of weights and numerators?);
  print £ volume = ?,sameline,v,££12? end of part one ?;
precompile;
lb2:
begin
comment ABSORPTION TWO;
  integer  h,k,l,ana,anb,anc,lrn,lwt,ran2,ran3,ran4,awt,
           arn,acf,lcf1,lcf2,lcf3,moda,modb,ha,ka,la,hb,kb,lb,
           hh,kk,ll,lp,length,absn,digh,digk,digl,lhs,rhs;
  real mu,calpha,cbeta,cgamma,astar,bstar,cstar,stheta,
       xoi,yoi,zoi ,xor,yor,zor,zeta,xi,has,kbs,lcs,cnu,
       cwa,cwb,cwc,cwd,cwe,cwf,cwg,cwh,cwi,
       swc,swd,swf,swg,swh,swi,lla,llb,llc,
       rasa,rasb,rbsa,rbsb,rd,i,q,scale;
  switch ss:=l1,l2,l3,l4,l5,l6,l7,l8,lorpol,newnet;
  Boolean ba;
  ran2:=range(rn,2);  ran3:=range(rn,3);  ran4:=range(rn,4);
  awt:=address(weight);  arn:=address(rn);acf:=address(coeffa);
newnet:
  print ££12??;  nn:=15;
  instring(title,nn);  nn:=15;  outstring(title,nn);  print££12??;
    punch(2);
read calpha,cbeta,cgamma,astar,bstar,cstar,moda,modb,
     ha,ka,la,hb,kb,lb,scale,absn,lp,length,digh,digk,digl,lhs,rhs;
  if absn = 1 then read mu;
  advance(1);  if not buffer(1,£*?) then
  begin
    print £ basic data error ?;  wait;  go to newnet;
  end;
```

```
 ba:=cbeta=0 and calpha =0;
 cwa:=checkr((calpha-cbeta*cgamma)/
      (sin(arccos(cbeta))*sin(arccos(cgamma))));
 cwb:=checkr(sin(arccos(cwa))*sin(arccos(cbeta)));
 if ba then
           begin  cwc:=checkr(1);  swc:=checkr(0)  end
 else
begin
     cwc:= checkr(cbeta/sin(arccos(cwb)));
     swc:= checkr(sin(arccos(cwc)))
end;
 advance(1);
 modb:=modb+4;
 if moda=1 then ll:=ha else if moda=2 then kk:=ha else hh:=ha;
 for ne:=la step 1 until lb do
begin
 if buffer(1,£*?) then  advance(1);
 for nf:=ka step 1 until kb do
 if  not buffer(1,£*?) then
begin
 read i;  advance(1);
 go to ss[moda];
 l1:  kk:=ne;  hh:=nf;  go to ss[modb];
 l2:  ll:=ne;  hh:=nf;  go to ss[modb];
 l3:  ll:=ne;  kk:=nf;  go to ss[modb];
 l4:  h:=hh;  k:=kk;  l:=ll;  go to l7;
 l5:  h:=ll;  k:=hh;  l:=kk;  go to l7;
 l6:  h:=kk;  k:=ll;  l:=hh;
 l7:
 if i = 0 then goto l8;
 has := h*astar;  kbs := k*bstar;  lcs:= l*cstar;
 lla:=checkr(lcs*sin(arccos(cwb)));
 if h=0 and k=0 then begin llb:=0; cwd:=1 ; swd:=0 end
 else
 begin
 llb:=checkr(sqrt(has↑2+kbs↑2+2*has*kbs*cgamma));
 cwd:=checkr((has+kbs*cgamma)/llb);
 swd:=checkr((kbs*sin(arccos(cgamma)))/llb);
 end;
 cwe:=checkr(-cwd*cwc-swd*swc);
 llc:=checkr(sqrt(lla↑2+llb↑2-2*lla*llb*cwe));
 if absn ≠ 1 then goto lorpol;
 stheta:=checkr(sqrt(has↑2+kbs↑2+lcs↑2+2*kbs*lcs*calpha+
         2*has*lcs*cbeta+2*has*kbs*cgamma)/2);
 cwf:=checkr(llc/(2*stheta));
```

```
  if cwf > 1 then cwf:=checkr(1);
  cwf:=checkr(cos(arcsin(cwf)));
  swf:=sin(arccos(cwf));
  cwg:=checkr((lla*cwc+llb*cwd)/llc);
  swg:=checkr((lla*swc+llb*swd)/llc);
  cwh:=checkr(cwf*stheta);
  swh:=sin(arccos(cwh));
  cwi:=checkr((stheta-cwf*cwh)/(swf*swh));
  swi:=sin(arccos(cwi));
  checks(£direction cosines?);
  xoi :=checkr(swh*(cwg*cwi+swg*swi));
  yoi :=checkr(swh*(swg*cwi-cwg*swi));
  zoi :=checkr(cwh);
  xor :=checkr(swh*(cwg*cwi-swg*swi));
  yor :=checkr(swh*(swg*cwi+cwg*swi));
  zor :=checkr(cwh);
  q :=0;
  for na :=1 step 1 until mx do
begin
comment SUMMATION;
  ana:=(na-1)*ran2;
  for nb :=1 step 1 until my do
begin
  anb:=(ana+nb-1)*ran3;
  for nc :=1 step 1 until mz do
begin
  anc:=(anb+nc-1)*ran4;
  lwt:=anb+nc-1+awt;
begin
  rasb:= rbsb:=0;
  for nd:= 1 step 1 until rowmxa do
begin
  lrn:=anc+nd-1+arn;
  lcf1:=4*(nd-1)+acf;
  elliott(0,2,lcf1,0,2,0,lcf2);
  elliott(0,2,lcf2,0,2,0,lcf3);
  checks(£rasa?);
  rd:=location[lcf1]*xoi+location[lcf2]*yoi+location[lcf3]*zoi;
  if rd ≠ 0 then rasa :=checkr(location[lrn]/rd)else rasa :=0;
  if rasb=0 and rasa > 0 then rasb:=rasa
  else if rasa > 0 and rasa < rasb then rasb:=rasa;
  checks(£rbsa?);
  rd:=location[lcf1]*xor+location[lcf2]*yor+location[lcf3]*zor;
  if rd ≠ 0 then rbsa:=checkr(location[lrn]/rd)  else rbsa :=0;
  if rbsb =0 and rbsa > 0 then rbsb :=rbsa
  else if rbsa > 0 and  rbsa < rbsb then rbsb := rbsa
end;
```

```
  checks(£q?);
  q :=checkr(q+location[lwt]*exp(-mu*(rasb+rbsb)));
end
end
end
end;
  q:=q/v;
  i := i/q;
 i := checkr(i);
  lorpol:
  xi := llc;  zeta := los*cwb;
  if lp = 1 then
  begin
  comment  LORENTZ AND POLARISATION FACTORS AND SPOT LENGTH
           CORRECTION;
  cnu := cos(arcsin(zeta/2));
  i := checkr(i*((xi/2)*sqrt(4*cnu↑2-xi↑2)));
  i := checkr(i*(8/(8-4*xi↑2-4*zeta↑2+xi↑4+2*xi↑2*zeta↑2+zeta↑4)));
  end;
  if length = 1 and 1 ‡ 0 then
  i := checkr(i*(1+((180*zeta*sqrt(-1+((4-zeta↑2)/xi↑2)))
       /(4*3.142*(75+(28.7/(sqrt(1-(zeta/2)↑2)))))));
  i := i*scale;
  i := sign(i)*sqrt(abs(i));
18:
  if i ‡ 0 then
begin
  if i > 0 then punch(1) else punch(2);
  print digits(digh),hh,sameline,£=?,digits(digk),kk,£=?,digits(digl),ll,
        £=?,aligned(lhs,rhs),abs(i),£=?;
  end;
end
end;
  advance(1);
  if not buffer(1,£F?) then goto newnet;
end
end
end
end;
```

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