# STRUCTURAL STUDIES IN TELLURIUM CHEMISTRY

by

ANTONY ALAN WEST

A Thesis Submitted for the Degree of Doctor of Philosophy

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## SUMMARY

The primary theme of this research was the characterisation of new and novel organo-tellurium complexes. Using the technique of single crystal X-ray analysis to establish more firmly the various coordination modes of tellurium. In each study the unit cell dimensions and intensity data were collected using an Enraf-Nonius CAD-4, four circle diffractometer. The raw data collected in turn was transferred to the Birmingham University Honeywell Multics System and processed using the appropriate computer packages for the determination of crystal structures.

The molecular and crystal structures of bis[2-(2-pyridyl)phenyl]tritelluride, bis[2-(N-hydroxy)iminophenyl] ditelluride, 2-(2-pyridyl)phenyltellurium(IV) tribromide, (2-N,N-dimethylbenzylamine-C,N')tellurium(IV)tribromide, 2-dichloro(butyl)tellurobenzaldehyde, 2-dichloro(butyl)telluro-N-dimethylbenzyl ammonium chloride, dimethyldithiocarbamato[2-(2-pyridyl)phenyl]tellurium(II), dimethyldithiocarbamato[2-(2-quinolinyl)phenyl]tellurium(II) and *para*-ethoxyphenyl[2-(2- pyridyl)phenyl]telluride are described. In each structure, the Lewis acidity of tellurium appears to be satisfied by autocomplex formation, through short-range intramolecular secondary bonds between tellurium and an electron denoting species, (generally nitrogen in these structures). With long range weak inter-molecular contacts forming in the majority of the tellurium(IV) structures. The order of Lewis acidity in each structure can be considered to be reflected by the length of the short range intramolecular secondary bond, identified, that is, when tellurium has a low Lewis acidity this interaction is long. Interestingly, no primary bonds are found *trans* to a Te-C covalent bond in any of the above structures, highlighting the strong *trans* effect of aromatic and aryl groups in tellurium complexes.

KEY WORDS: Organotellurium, X-ray crystallography, Lewis acidity, *trans* effect, Secondary bonding.

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# CHAPTER ONE

# INTRODUCTION

## 1.1 General Introduction and Theory of Single Crystal X-ray Crystallography

The study of molecules at the atomic level manifests itself in various forms, from simply defining a compound's molecular formula to revealing its quantitative structure. The latter provides the exact geometry between atoms, high-lighting regular and irregular arrangement of atoms, within the system. Single crystal X-ray crystallography is a technique that is widely used as a method of determining the three dimensional structure and molecular dimensions of inorganic and organic molecules. A well refined structure provides unambiguous information on the connectivity of the atoms of the molelcules in the solid state. This procedure is often used to confirm or refute or simply add to the information on structures proposed on the basis of synthetic pathways or analytical procedures such as mass spectrometry, nuclear magnetic resonance, elemental microanalysis and infrared and ultraviolet spectroscopy.

The characterisation of a single crystal by X-ray diffraction is based on the premise that the internal structure of the crystal is an infinite array of molecules, ions or atoms regularly repeating in three dimensions. This internal structural regularity enables the crystal to act as a three dimensional diffraction grating to X-rays, as the wavelength of X-rays is of the same order as the interatomic distances in the crystal. (Von Laue, Fredrick and Knipping, 1913). In the same year, Bragg and Bragg, solved the first crystal structure by considering the crystal as a three dimensional array of points arranged in planes from which

X-rays are reflected[1].

For a reflection to occur, the reflected rays must be in phase. The path difference between rays reflected from successive planes depends on d, the interplanar spacing which is a function of the unit cell and the angle of incidence θ. This in turn is equal to 2d sinθ. If the wave length of the X-rays is λ, this then leads to the Bragg equation:

$$n\lambda = 2d \sin\theta$$

From the intensities of the reflections it is possible using the method outlined below, to discover the positions of the individual atoms in the crystal and hence their stereochemical arrangement.

### 1.1.1 Scattering Factor

The scattering power or form factor of an atom is a function of its type and its finite size which varies with $\sin\theta/\lambda$. At $\sin\theta/\lambda=0$ the value of the scattering factor of a neutral atom is equal to its atomic number, since all electons scatter in phase. The fall-off in intensity with increasing scattering angle increases with the increase in the vibrations of the atoms, and these vibrations in turn increase with rising temperature. To account for this fact the form factor is multiplied by a temperature factor:-

$$f=f_0 e^{-B_{iso}[(\sin^2\theta)/\lambda^2]}$$

Where $B_{iso}$ is the temperature factor (Debye-Waller factor),

which is equal to $8\pi^2<U^2>$, where$<U^2>$ is the mean square amplitude of atomic vibration.

### 1.1.2 Structure Factor

The structure factor, F(hkl), (where hkl are Miller indices of the lattice plane) expresses the combined scattering of all atoms in the unit cell compared to that of a single electron. The components required for the combined scattered wave from the (hkl) planes are $f_j$ and $\phi_j$ where $f_j$ is the atomic scattering factor for the jth atom in the crystal corrected for thermal vibrations and $\phi_j$ is the phase angle of the scattering associated with the $j^{th}$ atom.

If there are N atoms in the unit cell then,

$$F(hkl) = \sum_{j=1}^{N} f_j e^{(i\phi j)} = \sum_{j=1}^{N} f_j e^{[i2\pi(hx_j+ky_j+lz_j)]} \qquad (3)$$

For reflections from successive planes of the set hkl there is a phase difference of $2\pi$ radians. Thus $\phi_j = 2\pi (hx_j+ky_j+lz_j)$, (as illustrated above) where $x_j$, $y_j$, and $z_j$ are the co-ordinates of the $j^{th}$ atom expressed as fractions of the unit cell.

The structure factor can be represented either exponentially or as a complex number thus:

$$F(hkl) = A(hkl) + i\,B(hkl) \qquad (4)$$

$$\text{where } A(hkl) = \sum_{j=1}^{N} f_j \cos 2\pi (hx_j + ky_j + lz_j) \qquad (5)$$

$$\text{and } B(hkl) = \sum_{j=1}^{N} f_j \sin 2\pi (hx_j + ky_j + lz_j) \qquad (6)$$

The amplitude of the wave is given by:

$$/F(hkl)/ = \sqrt{A(hkl)^2 + B(hkl)^2} \qquad (7)$$

and the phase angle $\phi_{hkl}$, associated with $F_{hkl}$ is given by:

$$\tan \phi(hkl) = \frac{B(hkl)}{A(hkl)} (8) \quad \text{and } F(hkl) = /F(hkl)/e^{i\phi(hkl)} \qquad (9)$$

### 1.1.3 The Fourier Synthesis

The general electron density function is expressed as a three dimensional Fourier series, in which the number of electrons per unit volume or electron density at any point x,y,z, represented by $\rho(x,y,z)$ is given by:

$$\rho(x,y,z) = \frac{1}{V_c} \sum_{\text{all hkl}} \sum \sum F(hkl) e^{-2\pi i(hx+ky+lz)} \qquad (10)$$

where $V_c$ = Unit Cell Volume.

In principle hkl range from $-\infty$ to $\infty$, however the summations in practice extend over a finite set of hkl, which are limited by both the nature of the crystal and the particular pre-set experimental limits. These factors lead to errors in the electron density map, and are known as termination of series errors. Substituting $/F(hkl)/e^{i\phi(hkl)}$ for F(hkl) and taking

summations over the appropriate practical values of h, k and l.

$$\rho(x,y,z) = \frac{1}{V_c} \sum_{h} \sum_{k=-\infty}^{\infty} \sum_{l} /F(hkl)/\cos\,[2\pi(hx+ky+lz)-\phi(hkl)] \qquad (11)$$

Assuming Friedel's law holds, the sine terms cancel out in pairs of

$$/F(hkl)/ \text{ and } /F(\overline{hkl})/ \qquad (12)$$

to give:

$$\rho\,(x,y,z) = \frac{2}{V_c} \sum_{h=0} \sum_{k=-\infty}^{\infty} \sum_{l} /F(hkl)/\cos\,[2\pi(hx+ky+lz) - \phi(hkl)] \qquad (13)$$

Since the maximum necessary summations now take over a hemisphere in reciprocal space. The term F(000), which represents the total number of electrons in the unit cell, is multiplied by $1/V_c$ and in practice; the term $F(000)/V_c$ is added separately to the result of the summations.

If we knew /F(hkl)/ and ø, for each h, k, l it would be possible to compute the electron density for all of the values of x,y,z. These values could in turn be plotted to give a three dimensional electron density map. Unfortunately, however the phase angles ø are not a directly measurable quantity and this phenomenon thus consititutes the so called phase problem of X-ray crystallography.

### 1.1.4 Data Reduction

Since the intensity of any radiation propagated as a wave is proportional to the square of its amplitude, the intensity of the

diffracted beam corresponding to the diffraction maximum hkl is proportional to the amplitude squared, except for geometric factors, absorption corrections (if needed), and other minor effects. Assuming absorption effects to be negligible, this may be expressed thus:

$$/F(hkl)/=(KI(hkl)/Lp)^{1/2} \qquad (14)$$

Where p is the polarization factor, which arises because the reflecting efficiency of the X-ray beam varies with $\theta$, and is given by:

$$p= (1 + \cos^2 2\theta)/2 \qquad (15)$$

and L is the Lorentz factor, which expresses the fact that, for a constant angular velocity of rotation of the crystal, different reciprocal lattice points pass through the sphere of reflection at different rates and thus have different times of reflection opportunity. The form of the Lorentz factor depends upon the experimental arrangement. For both zero level photographs taken with the X-ray beam normal to the rotation axis and four-circle diffractometer measurements, L has the form of:

$$L= 1/\sin 2\theta \qquad (16)$$

K, the scale factor can be determined from statistical procedures , ( The Wilson Method )[2]. It depends on the beam

intensity, crystal size and other fundamental constants. It is the same for a given set of measurements and thus is omitted, and relative scale factor are calculated, such that:

$$/F_{rel}/ = K/F_o/ = (I(hkl)/Lp)^{1/2} \qquad (17)$$

The scaling between $/F_{rel}/$ and the observed structure amplitude $/F_o/$ is obtained by comparison of $/F_{rel}/$ with the calculated structure factor amplitude $/F_c/$ at a later stage of the structure determination.

### 1.1.5 The Patterson Synthesis

The lack of knowledge concerning the phases of the structure factors prevents us from directly computing the positions of the atoms in the unit cell. In 1934, A L Patterson, defined a function, which circumvented this difficulty, as it required no phase information(3).

$$P(u,v,w) = V \int_0^1 \int_0^1 \int_0^1 \rho(x,y,z)\,\rho(x+u,y+v,z+w)\,dx\,dy\,dz \qquad (18)$$

by substituting the values for the electron density obtained in equation 10 into equation 18 and simplifying it can be shown that;

$$P(u,v,w) = 1/v \sum_h \sum_k \sum_l /F(hkl)/^2 \exp[2\pi i(hu+kv+lw)] \qquad (19)$$

The Patterson function is a Fourier series with zero phases

and the squares of the structure amplitudes as coefficients. The form of the expressions which is usually used is:

$$P(u,v,w) = 1/v \sum_h \sum_k \sum_l |F(hkl)|^2 \cos 2\pi (hu + kv + lw) \quad (20)$$

It can be seen from equation 10 that P (u,v,w) is a maximum when there is high electron density at both x,y,z, and x+u, y+v, z+w, thus information about interatomic vectors can be gained from a Patterson series.

The weight of the Patterson peak depends upon the number of electrons in the atoms between which the vectors occur. If there are one or a few atoms of high atomic number present within the structure, the largest maxima will correspond to vectors between these atoms. This means it is possible to calculate the positions of the heavy atom(s) from the knowledge of the symmetry of the unit cell. In particular it has been shown that if a crystal contains axes or planes of symmetry, then the vectors joining symmetry related atoms appear as peaks on planes and lines in the Patterson synthesis[4]. The location of the heavy atom(s) provides a means of calculating the phases of the waves using the structure factor equations. This is based on the premise that the phases may be approximated to the phases of the heavy atom(s) initially.

$A(hkl) = f^H \cos 2\pi(hx^H + ky^H + lz^H) + \sum f^{LA} \cos 2\pi (hx^{LA} + ky^{LA} + lz^{LA})$

where $f^H$ = atomic scattering factor of the heavy atom and $f^{LA}$ =

atomic scattering factor of the lighter atoms.

$B(hkl)=f^H \sin 2\pi(hx^H+ky^H+lz^H) + \Sigma f^{LA} \cos 2\pi (hx^{LA}+ky^{LA}+lz^{LA})$

Because $f^H >> f^{LA}$ we can gain an approximate phase for the structure by ignoring the scattering of the lighter components.

$$\phi^H (hkl) = \tan^{-1} \frac{B^H (hkl)}{A^H (hkl)}$$

The electron density map produced using the experimental structure amplitudes and calculated phases, should reveal most if not all of the lighter atoms within the structure. The location of the lighter atoms allows the recalculation of the phases from the structure factor equations, giving a better approximation to the 'true' phase.

A point to bear in mind initially, is that, in a structure with N atoms per unit cell, each atom forms a vector with the remaining N-1 atoms. There are, thus, N(N-1) non origin peaks representing the inter-atomic vectors. The Patterson unit cell is the same size and shape as the crystal unit cell, but it has to accommodate $N^2$ rather than N "peaks" and is therefore correspondingly overcrowded. Peaks in Patterson space tend to overlap when there are many atoms in the unit cell, a feature which introduces difficulties into the process of unravelling the functions in terms of the correct distribution of atoms in the crystal. It is often useful, therefore to modify this synthesis by means of a sharpening process in which atoms are considered to have all their scattering power concentrated at the nucleus i.e. point atoms. The approximation used is of the form,

$$/F_{mod}(hkl)/^2 = \frac{/F_o(hkl)/^2}{\exp\ [-2B(\sin^2\theta)/\lambda^2]\{\sum_{j=1}^{N} f_j\}} \qquad (21)$$

This procedure is the "classical" heavy atom method for structure determination. If the structure does not contain a heavy atom, it is still theoretically possible to solve the structure from a Patterson synthesis. Although this is usually extremely difficult and so recourse is made to the alternative procedure called direct methods.

### 1.1.6 Direct Methods

It is possible to derive relations among the phases of different reflections. These relations come from the fact that the electron density can never be negative and that it is near zero except for isolated resolved peaks at atomic positions. For centrosymmetric structures, only signs of + or - are involved because cos $\alpha$ = +1 or -1 and sin $\alpha$ = o, as the phase associated with each amplitude is limited to 0 or 180°.

The type of structure factor generally employed in this method of analysis, is the normalized structure factor. This factor provides a method for simplification whereby /F(hkl)/ is substituted by the corresponding /E(hkl)/ value, thus:

$$/E_o\ (hkl)/^2 = \frac{K^2/F_o(hkl)/^2}{\varepsilon \sum_{j=1}^{N} f_j^2} \qquad (22)$$

where the $\varepsilon$-factor takes account of the fact that reflections in certain reciprocal lattice zones or rows may have an average intensity greater than that for the general reflections. The $\varepsilon$ -factor depends upon the crystal class. K is the scale factor for the $/F_o(hkl)/$ data.

In 1952 Sayre derived a general formula for structures containing resolved atoms, for centrosymmetric crystals[5]. For three large reflections, hkl, $h'k'l'$ and $h-h'$ $k-k'$ and $l-l'$.

$$s(hkl)s(h'k'l')s(h-h', k-k', \text{and } l-l') \approx +1 \qquad (23)$$

where the sign $\approx$ means "is probably equal to" and s(hkl) means the sign of F(hkl). Cochran[6] and Zachariasen[7] also arrived at this probability relationship although by different lines of reasoning.

A more general form of this equation was given by Hauptman and Karle ($\Sigma_2$ Formula)[8].

$$s[E(hkl)] \approx s\left[\sum_{h'k'l'} E(h'k'l')E(h-h', k-k', l-l')\right] \qquad (24)$$

The summation being over all vectors pairs with known signs which form a triplet with hkl. The probability P that this equation will hold is given by:

$$P_+(hkl) = 1/2+1/2\tanh[(\theta_3/\theta_2{}^{3/2})\alpha'] \qquad (25)$$

where $\alpha'$ is given by:

$$\alpha' = /E(hkl)/\sum_{h'k'l'} E(h'k'l')\ E(h-h', k-k', l-l') \qquad (26)$$

and $\theta_n$ by:

$$\theta_n = \sum_j Z_j^n \qquad (27)$$

where $Z_j$ is the atomic number of the $j^{th}$ atom. If all the N atoms of the cell are equal, it can be shown that:

$$\theta_3/\theta_2^{3/2} = 1/\sqrt{N} \qquad (28)$$

and the expression for P becomes,

$$P_+(hkl) = 1/2 + 1/2\tanh[(1/\sqrt{N})\alpha'] \qquad (29)$$

The symbolic addition method, forms the basis for structure determination[9]. Initially, a small number of reflections, usually three (less in the case of a non-primitive lattice), can, subject to restrictions depending on the space group of the crystal be assigned arbitrary phases. These choices determine which of the several equivalent positions is used for the origin of the unit cell. The three sets of h,k,l, must be different with respect to the evenness or oddness of indices, and h,k,l must not all be even. In addition, several other reflections which take part in a reasonable number of $\Sigma_2$ relationships are assigned symbolic or actual numeric phases[10]. It is these signs which form a basic set to which a "snow-balling" technique allows more and more reflections to be included as the analysis proceeds. An E map, a Fourier synthesis where the coefficients are Es instead of Fs can be computed for each phase set in order of probable correctness until a reasonable set of atomic positions is found.

An alternative centrosymmetric approach using direct methods, is that adopted in the SHELX computer packages[11,12], used in this study. In this analysis, rather than using the recycling technique, a very large number of permutations of signs (of the order of $2^{10}$ to $2^{20}$) are initially computed. Those sets which give a poor agreement, during sign expansion are eliminated. The prescribed level for agreement, in turn increases during the expansion. E-maps are computed and figures of merit are calculated for the surviving sets. The initial figure of merit used, should be a minimum for the most probable phase set :-

$$\text{ABS Fom} = \frac{\sum_h \alpha_h - \sum_h \langle \alpha^2_r \rangle^{1/2} r}{\sum h \langle \alpha^2_h \rangle^{1/2} e - \sum_h \langle \alpha^2 \rangle^{1/2} r} \qquad (30)$$

where:

$$\alpha_r = \sum_h \left( \sum_{h'} K^2_{hh'} \right)^{1/2} \qquad (31)$$

which is the value of $\alpha_h$ assuming random phases and:

$$k_{hh'} = 2\sigma_3 \sigma_2^{-3/2} / E_h E_{h'} E_{h-h'} / \qquad (32)$$

The second figure of merit,

$$P+ = 1/2 + 1/2 \tanh 1/2 N / E_{h-h'} E_{h+h'} / (2E^2_h E^2_{h'} - E^2_h - E^2_{h'}) \qquad (33)$$

which is based on Giacovazzo's work on quartet relationships[13].

Direct methods has been extended to non-centrsymmetric crystals, however their discussion is omitted from this work as the technique was not employed.

### 1.1.7 The Difference Synthesis

It is not always possible to identify all the atomic positions from an E-map or from a heavy atom Fourier. To circumvent this difficulty, the method of difference synthesis is invoked to locate the positions of the remaining atoms. This type of synthesis is a Fourier Series whose coefficients are $\Delta F$,(where $\Delta F = /F_o/ - /F_c/$) in place of $/F_o/$[14]

A map calculated in this manner eliminates series termination errors, as they are present in both $\rho_o$ and $\rho_c$. Random errors do produce small fluctuations in a difference map, but these should be within two and a half to three times the standard deviation of the electron density $\sigma(\rho_o)$,

$$\sigma(\rho_o) = \frac{1}{VC}\left[\sum_{hkl} (\Delta F)^2\right]^{1/2} \qquad (34)$$

At a later stage of the structural refinement, this technique may be applied to determine the positions of the hydrogen atoms. After the determination of the complete structure the difference map should be essentially featureless.

### 1.18 Refinement

Since there are many more observations than parameters to be determined, statistical methods can be used to fit an appropriate equation expressing the errors in /F/ that result from the errors in the trial structure. The method of refinement

used to determine the atomic co-ordinates and temperature factors with the greatest possible accuracy is based on finding the best fit between the observed and calculated structure amplitudes using the principle of least squares. Where the quantity to be minimised is,

$$D = \Sigma_{hkl} W_{hkl} (/F_o/ - /KF_c/)^2 \qquad (35)$$

Where $\Sigma_{hkl}$ indicates the summation over all of the observed reflections, K is a scaling factor and $W_{hkl}$ is the weight of the observation. The weighting scheme used is chosen to give the smallest variation of the mean value of $W_{hkl}$ $(/F_o/ - /F_c/)^2$ as a function of $/F_o/$ and $\sin \theta/\lambda$.

Refinement is a cyclic procedure, revealing at the end of each cycle various discrepancy indices along with the shift/esd for each parameter. The agreement between $F_o$s and $F_c$s can be described by the residual or reliability index R, which decreases as the refinement proceeds.

$$R = \frac{\Sigma / /F_o/ - /F_c/ /}{\Sigma /F_o/} \qquad (36)$$

This value is widely used, although it is not a perfect guide to the correctness of fit. A more useful term when comparing R values at different stages of refinement, is the weighted R value, $R_W$.

$$R_w = \sqrt{\frac{\Sigma W_i(/F_{oi}/ - /F_{ci}/)^2}{W_i/F_{oi}/^2}}$$

Where $W_i$ is the weight given to the $i^{th}$ observation.

The refinement is terminated when there is no improvement in the discrepancy indices and the shift/e.s.d.s are at an acceptable level for the various parameters i.e. at convergence.

### 1.1.9 Thermal Parameters

In the initial stages only one thermal parameter, the isotropic temperature $B_{iso}$, is considered. This implies a spherically symmetrical atom as was assumed in equation(2) for the scattering factor $f$. In reality the thermal vibration of the atom is not the same in every direction and allowance is made for this anisotropic vibration in the latter stages of refinement. The equation for $f$ then becomes:

$$f=fo \exp [1/4(B_{11}h^2a^{*2}+B_{22}k^2b^{*2}+B_{33}l^2c^{*2}+2B_{12}hka^*b^* +2B_{13}hla^*c^*+2B_{23}klb^*c^*)] \quad (38)$$

which can be expressed as

$$f=fo \exp [-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^* +2U_{13}hla^*c^*+2U_{23}klb^*c^*)] \quad (39)$$

where $U_{ij}$ are the thermal parameters expressed in terms of mean square amplitudes of vibrations in $Å^2$.

### 1.1.10 Limitations of Single Crystal X-ray Crystallography

Undoubtedly , crystallography provides information at a

molecular level which is unobtainable by other conventional analytical procedures. There are, however, weaknesses which limit the scope of X-ray crystallography.

Liquids and gases lack three-dimensional order, and cannot be used in diffraction experiments in the same way as crystals can. Therefore, obviously, before an examination of a structure by single crystal X-ray crystallography can take place, a single crystal of the compound needs to be obtained.

In general, bond lengths determined by X-ray methods represent distances between the centres of gravity of the electron clouds, which may not be the same as the internuclear separations. In addition, bond lengths and angles need to be interpreted with due consideration to the precision and overall accuracy of the determination. It may be difficult to distinguish unambiguously atoms of similar electron density, eg nitrogen atoms from carbon atoms in a heterocyclic ring. Although here, both routine analytical procedures and bond lengths may be of considerable aid in interpreting the results.

Hydrogen atoms are particularly difficult to locate with any precision because of their small scattering power, and the fact that the centre of the hydrogen atom does not, in general coincide with the centre of its electron density. Given high-quality intensity data, hydrogen positions can be expected to appear in a Difference Fourier Synthesis with a maximum electron density in the range 0.3 to 0.4 $e\text{Å}^{-3}$, but they may well be above or below these limits. Poor quality data or large thermal parameters or indeed a combination may make hydrogen positions unobservable.

## 1.2 General Introduction on Tellurium

Tellurium makes up approximately $10^{-9}$% of the earth's igneous rock. It was discovered in 1782 by Franz Joseph Muller von Reichenstein, although the actual element was not isolated and characterised until 1798 by Martin Heinrich Klaproth. Klaproth named the element Tellus or Earth after man's "heavenly body". Twenty-one isotopes of tellurium are know currently, with atomic masses ranging from 115 to 135. Of these isotopes only eight are found in naturally occuring tellurium. In today's periodic table, tellurium is located as the fourth element down in group 6A (or 16), having an atomic weight of 127.60 and an atomic number of 52.

Tellurium and tellurium complexes are used in a variety of ways in the world today. For example, tellurium is used as a brightener in electro-plating baths, [15] as an additive to copper and stainless steel to improve their machinability, it is added to lead to decrease the corrosive action of sulphuric acid and is also added to cast iron for chill control [16]. Tellurium also forms a basic ingredient in blasting caps, is an additive to catalysts for the cracking of petroleum and is used as a colourant for glasses. Interestingly, tellurium is a p-type semi-conductor which shows greater conductivity in certain directions depending on the alignment of the atoms[15,16].

Organotellurium chemistry forms a growing and interesting part of organometallic chemistry, which is flourishing at present.

Historically, the first organotellurium compound was synthesized in 1840 by Wohler, since that time, the field of organotellurium chemistry has grown steadily to include telluromercaptans (RTeH), five and six membered cyclic tellurides, telluronium compounds ($R_3TeX$), tellurinic acids (RTeOOH), telluric esters [$(RO)_6Te$], telluroketones ($R_2CTe$), telluroxides ($R_2TeO$), tellurenes ($R_2TeO_2$), dialkyltellurides ($R_2Te$), dialkyltellurium dihalides ($R_2TeX_2$) and alkyl- and aryl- tellurium trihalides ($RTeX_3$). Throughout this century, the literature on this topic has grown [17-20], with a number of noteable reviews appearing in the last couple of decades.[21-23]

This 'explosion' of interest in organotellurium chemistry and the associated development in the co-ordination chemistry of tellurium, has lead to the incorporation of organotellurium compounds into a number of interesting areas. In an early review by Gysling, two kinds of process were described using organotellurium compounds or their complexes as imaging systems in photography[24]. A limited number of publications have described the use of tellurium compounds or their complexes as reagents for organic synthesis[25]. In addition, organochalcogens containing one or two chalcogen-chalcogen bonds when coordinated with transition metal ions have been used as active materials for battery electrodes[26]. Materials of this type have also shown significant conductivity in the solid state [27].

### 1.2.1 Work at Aston University, in the Speciality Materials Group

Interest in the field of Organotellurium Chemistry has been established over the last two decades at Aston University. Latterly this interest has concentrated on the synthesis and properties of some novel tellurium containing ligands[28-30] and on the charge transfer in organotellurium compounds and their complexes[31-33]. The long term goal of the group is the design of molecular conductors, which are formed from new organotellurium ligands which wrap themselves around a central metal ion to give a square planar geometry. This aim is obviously some way into the future.

This rationale for using organotellurium ligands for this purpose is based on the premise that tellurium is a large and easily polarisable element, which forms complexes with metal ions which have soft acid character[22,34] as tellurium itself is a soft base. The assignment of soft acid and soft base, stems from the theory put forward in 1923 by G N Lewis. He stated that substances which are capable of acting as ligands, are bases and the substances that they react with, are acids. Put another way, this means a Lewis base is a donor of an electron pair and a Lewis acid is an acceptor of an electron pair[35]. The terminology of hard and soft, was preposed by Pearson in 1963[36,37], who defined hard bases, as having high electronegativity, a low polarisability and a high resistance to oxidation, whereas soft bases have a low electronegativity, a high polarisability and are readily oxidised. Soft acids on the other hand, were defined as

large, of low charge or having valence shell electrons, which are easily distorted or removed. This means, this type of acid generally prefers to bond to soft bases, whereas hard metal ions (acids) usually bond to hard ligands (bases).

### 1.2.2 Classification of Organotellurium Compounds

Organotellurium compounds can be classified broadly into four major groups which have the following general formulae:

(1) $R_2TeX_{2-n}$ (n=1 or 2)

(2) $R_2Te_2$

(3) $R_nTeX_{4-n}$ (n=1,2,3,or 4)

(4) Heterocyclic tellurium compounds :

- (i) Five-membered ring systems
- (ii) Six-membered ring systems
- (iii) Six-membered ring systems with either O, S or Se

Where R may be alkyl or aryl groups and X ranges from electronegative groups such as OR, OCOR, halogen, pseudohalogens, other acid radicals through neutral ligands such as H to electropositive atoms such as Li or Na.

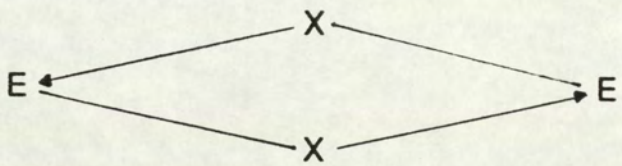
### 1.2.3 Lewis Acidity of Organotellurium Compounds

In this study we are concerned solely with the Lewis acid behaviour of tellurium, within organotellurium ligands. In such a

system, tellurium can act as a Lewis acid to "complex", with a neutral Lewis base or an anionic one, via simple complex formation.

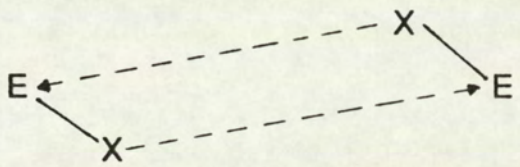
$$R_n EX_{4-n} + XL \ldots\ldots\ldots\ldots R_n EX_{4-n}\cdot(L)_X$$

or alternatively, the Lewis acidity of tellurium may be satisfied by auto-complex formation[38]. Here the organotellurium ligand is amphoteric in the Lewis sense, as centres of Lewis acidity and basicity are present within the same molecule. Auto-complex formation may then occur between these centres either via inter- or intra- molecular interactions. The most common examples of short range intermolecular interactions occurs when a halogen bridges two acid centres. The implication of this type of arrangement, is that the components of the bridge bond are approximately the same.

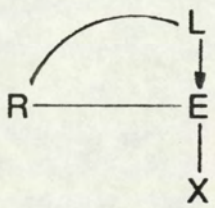



The second type of auto-complex formation involves long-range intermolecular interactions, where one set of E---X bonds is significantly longer than the sum of the covalent radii of E and X, but shorter than the van der Waals distance. Alcock in his 1972 review proposed a theory which explained this type of interaction, which he termed "secondary bonding"[39]. In this type of interaction, a secondary bond between E and X would be formed

via a dative donation of a lone pair of electrons from X to the antibonding ($\sigma^*$) orbital formed by the R-E bond, thus a linear arrangement would give rise to the most efficient bonding. (This theory is analogous to that which is generally accepted in hydrogen bonding.).




The final type of auto-complex formation is dubbed intramolecular complex formation, this occurs when it is sterically feasible for the acidic and basic centres to interact within the molecule: This situation is particularly prevalent when an organic group possess a donor group. Probably the most common example of this type of interaction is where an aryl group carries a basic function in a position *ortho* to E.




(In each category
E = tellurium
X = is an anionic group, often halogen
R = alkyl or aryl groups)

In both types of complex formation, the term "complex" denotes a product which has arisen from the reactions between a Lewis acid and Lewis base.

The question of the order of Lewis acidity within organo-tellurium complexes, was addressed by McWhinnie and

Monsef-Mirzai in their review entitled the "Co-ordination in Organoselenium and Organotellurium Chemistry" presented at the fourth international conference on the organic chemistry of selenium and tellurium[40]. They pointed out that the Lewis acidity of tellurium becomes less as more organic groups are attached to the metal. Thus for a series of Te(IV) halides (X), the order of Lewis acidity is:

$$RTeX_3 > R_2TeX_2 > R_3TeX$$

and for Te(II) the order is:

$$RTeX > R_2Te$$

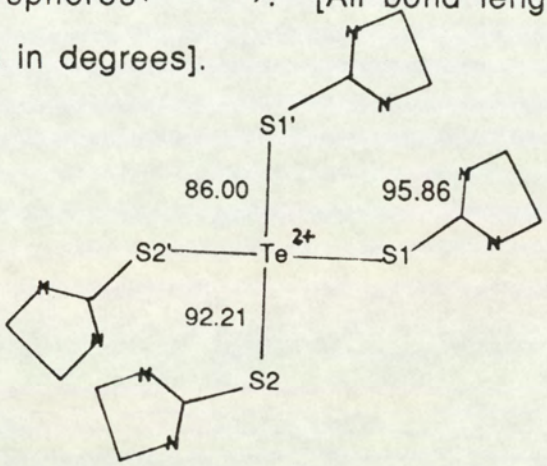
These scales although somewhat intuitive, relate mainly to the number of occurances where simple complex formation has been described.

### 1.2.4 Coordination of Tellurium in Oxidation States (II) and (IV)

From the early structural work on Te(II) complexes, it became clear that a square planar geometry seemed to be preferred.[41,42]. In this environment two of the p orbitals of the dipositive tellurium, each participate in linear three-centre bonding, so that two such systems are formed at right angles.

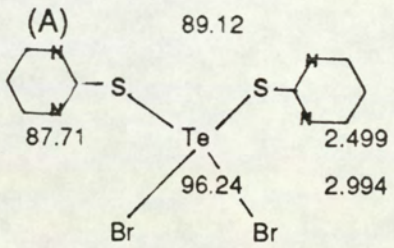
This preference for tellurium(II) complexes to adopt a square planar geometry became manifest from the structural work of Foss and co workers [41,42]. In four coordinate tellurium(II) complexes with monodentate sulphur and selenium ligands, square planar complexes are formed with $TeS_4$, $TeS_2Se_2$ and $TeSe_4$

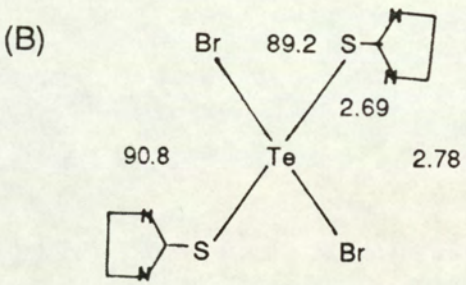
coordination spheres(43-57). [All bond lengths quoted are in Å's and all angles in degrees].




The tetrakis (ethylenethiourea) tellurium(II) cation in the complex salt $[Te(etu)_4][TeCl_6]$(44)

In square planar complexes with $TeS_2X_2$ and $TeSX_3$ coordination spheres, the three-centred four-electron bonding is highlighted by the relatively long tellurium to ligand bonds.

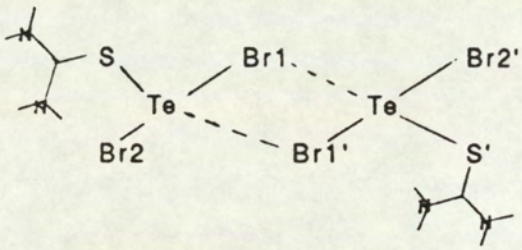



The structures of *cis*-dibromobis(trimethylenethiourea) -tellurium(II), (A), and *trans*-dibromobis(ethylenethiourea) tellurium (II), (B)(58,59).

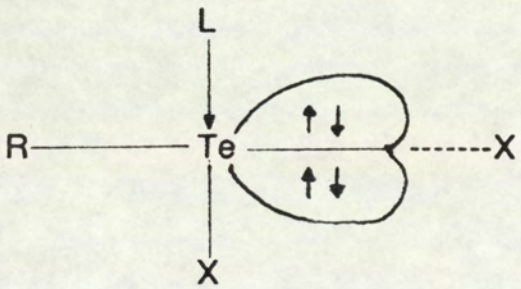
In the structures where a $TeSX_3$ coordination sphere appears, a distorted square planar coordination is obtained through inter-molecular Te--------X bonding which is analogous to the

corresponding selenium compound(60)




Two molecules of dibromotetra-methylthiourea tellurium(II)

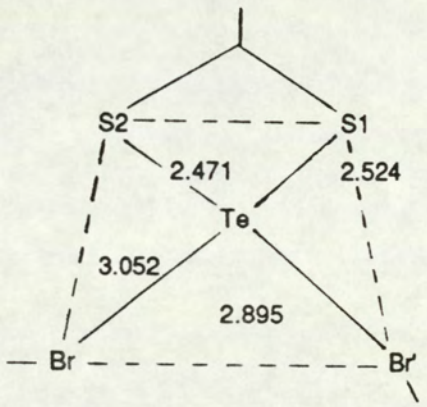
Naturally, this is only a tendency and not a rigid rule as a significant number of tellurium complexes are known where the central tellurium atom is three co-ordinate(61-67). In this environment, the molecular structure can be generally considered as "T" shaped, in which five electron pairs (two of which are non-bonding) distribute themselves around the tellurium atom to define a ψ trigonal bipyramid(40).




In about one third of the complexes, with this type of molecular structure, the fourth position in a square planar

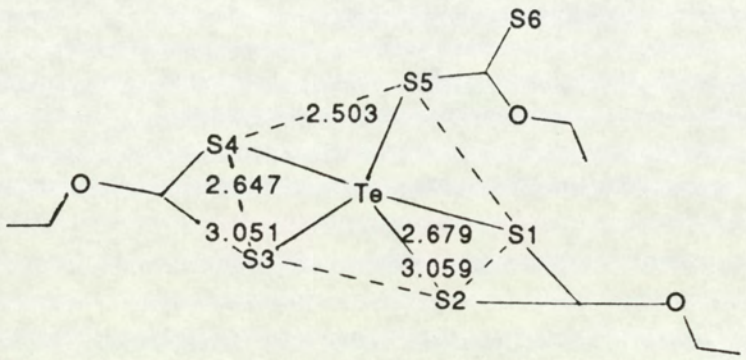
arrangement around tellurium is approached by a fourth ligand[61-63,65,66]. This phenomenon reinforces the view that the preferred geometry around tellurium(II) is square planar.

In four co-ordinate tellurium(II) complexes, with bidentate sulphur ligands a number of structures are known where the coordination sphere is greatly distorted[68-75]. In this type of system, tellurium is bound to two short Te-S bonds *cis* to each other, and roughly *trans* to these two weak bonds are formed. The resulting coordination around tellurium is a trapezoid, and trapezoid planar is a more correct description of the environment around tellurium in such cases[73,75].



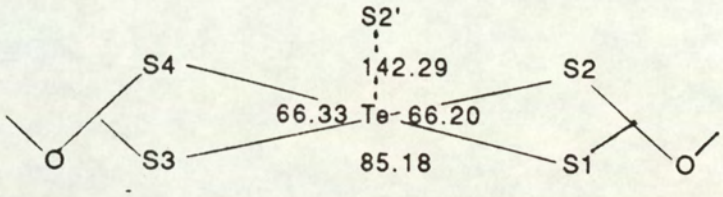

Polymeric bromo(ethylxantatho) Tellurium(II), with the central $TeBr_2S_2$ coordination sphere.

The structure of the tetraethylammonium salt of tris(ethylxanthato)tellurate(II) was reported by Hoskins and Pannan in 1975[76]. In this structure the tellurium is coordinate to two ligands in an anisobidentate manner and to the third ligand in a monodentate way. The resulting $TeS_5$ coordination sphere is pentagonal planar, thus in this complex tellurium(II) is five-coordinate.




The structure of tris-(ethylxanthato)tellurate(II)anion in its tetraethyl-ammonium salt.

There is a slight tendency towards pentagonal planar five coordination in tellurium(II) complexes, where tellurium is linked by a strong and a weak bond in an anisobidentate way, to the same ligand. When tellurium(II) is bonded to four sulphur atoms, in this way, a weak fifth intermolecular Te-S contact forms (3.5-3.7Å)[70].




Bis(methylxanthato)tellurium(II)

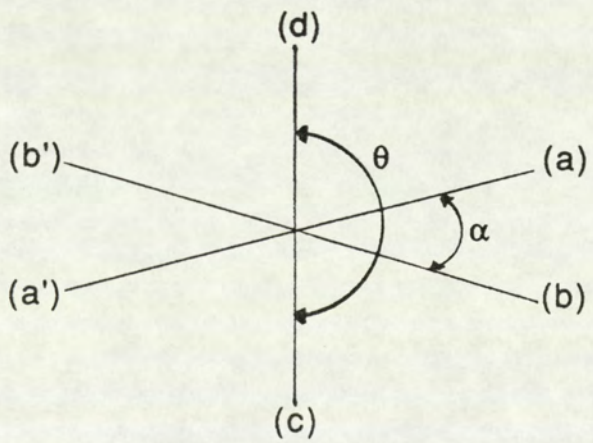
Te-S(1), 2.570 Å, Te-S(2). 2.841 Å, Te-S (3) 2.499 Å, Te-S(4) 2.846 Å, Te-S(2) 3.513 Å.

The coordination around tellurium in tellurium(IV) complexes seems to obey the localized electron pair (or VSEPR) theory of Gillespie and Nyholm[77,78], up to the coordination number five. According to this theory, the arrangement of bonds around any .one atomic centre depends on the number of electron

pairs surrounding that atom, and since each electron pair occupies one localized molecular orbital it depends on the number of localized molecular orbitals in which the atom participates and on the relative sizes and shapes of these orbitals. The preferred arrangment of a given number of electron pairs in the valency shell of an atom is that which minimises their repulsion energies.

The recognition that ligand atoms will distribute themselves on a coordination sphere is consistent with VSEPR theory, but the frequent lack of any geometric indication of the presence of electron pairs in the coordination sphere also seems to be well documented. When will an electron pair be inert in the stereochemical sense, and when will it not be? For coordination numbers higher than five, the lone pair of electrons ($5S^2$) in the valency shell of Te(IV) is mostly found to be stereochemically inert, in the sense that it does not occupy a position in the polyhedron.

In a review by Mangion, Zingaro and Meyers, entitled the "Stereochemistry of Tellurium"[79], it was concluded that the common coordination of atoms around Te(IV) in crystals is given by:



where, a-d are "normal" covalent bonds and a',b' are generally much shorter than van der Waal's contact and can be considered as weak secondary bonds[39].

The key word in this conclusion is common, it is not a certainty. Obviously there will be structures which deviate considerably from this "norm". Secondary interactions in different tellurium(IV) compounds give rise to differing overall structures, according to the significance which has been attributed to the inter- and intra- molecular associations.

In diorganotellurium(IV) dihalides for instance, the generally reported and therefore expected, primary structure is $\psi$ trigonal bipyramidal. Yet secondary interactions in different $R_2TeX_2$ structures have variously been assigned to structures described as distorted trigonal bypyramidal, distorted tetrahedral and distorted octahedral[80-87], depending on the importance assigned to the intermolecular associations through bridging halogens and the mixing of s with p electrons. In such systems, it is sometimes difficult to give enthusiastic support to the idea that the "normal" primary coordination around tellurium is trigonal-bipyramidal, when the "normal" value for the angle $\psi$ between the equatorial bonds is 120°, has been distorted to values ranging from 90.7(3) to 99.0(3)°, whilst the angles between the axial and equatorial bonds remains close to 90°[80,81,84.86,87].

Similar problems seem to arise in many other structures, than those sited above. It is never the less consoling to know, that the difficulties encountered when interpreting a structure in

the solid state are universally experienced.

The behaviour of Te(IV) when coordinated by S is more complicated than the common coordination of atoms around Te(IV) cited by Mangion et al[79]. A number of Te(IV) structures, where Te(IV) is coordinated by S fit this model reasonably well[88-91], but tris(diethyldithicarbamato)phenyltellurium(IV) in common with other $[Te(R_2NCS_2)_3Y]$ compounds (Y being Cl, Br, SCN and Ph) forms a seven coordinated pentagonal bipyramid[92-97]. Moreover, two eight-coordinated distorted dodecahedral structures are known[92,95,98-100]. The coordination in these structures is fairly difficult to represent in three dimensions, but may be visualised as two interleaving planar trapezoids at right angles to one another.

Tellurium exhibits a variety of coordination geometries which rest along a line of increasing occurrence. When considering the environment around tellurium, each alternative environment should be judged on its merits. It should not be assumed that the more common coordinations will be naturally adopted.

### 1.2.5 Objectives and Background of this Study

The aim of this study, is the identification and or authentication of unexpected, intermediate or final reaction products of new and novel tellurium ligands, by single crystal X-ray crystallography. It is anticipated that this analysis will establish more firmly the various coordination modes of

tellurium. Particular interest will be centred on the Lewis acid-base autocomplex formation, brought about by secondary bonding between tellurium and a ligand containing a donating atom or group.

Interest in this area at Aston University can be traced back, to the work of A. Z. Al-Rubaie, who examined the ability of organotellurium compounds to act as an electron donor, in charge transfer complexes involving N-methylquinolinium tetracyano-*p*-quinodimethane[31]. This theme of donor-acceptor complexes, involving organotellurium complexes was subsequently taken up by the work of H B Singh[32]. He reported the crystal and molecular structure of dibenzotellurophene-7,7,8,8-tetracyano-*p*-quino -dimethane. In this donor-acceptor complex, a significant intermolecular interaction is formed between stacks, through a short tellurium-nitrogen contact. [Te...N(2)(0.5-X, -1.5+Y, 0.5-Z) at 3.216(6) Å][32,101].

The synthesis and crystal structure determination of (2-phenylazophenyl-C,N)tellurium(II) chloride, highlighted an approach in designing new organotellurium donors, where the $\pi$ electrons are delocalised over a heterocyclic ring[102]. In this $\pi$ molecule an intramolecular Te...N contact occurs at 2.230 Å.

This approach has been extended by the work of M. A. K. Ahmed of the the speciality materials group at Aston University, who examined the structures of the azobenzene derivatives of tellurium(II) and tellurium(IV) complexes with monodentate and bidentate ligands. From this study, it is apparent that the environment in which the central tellurium atom is located has a

direct effect on the length of the tellurium-nitrogen interaction. Such that the length of the tellurium-nitrogen interaction appears to depend on the oxidation state of tellurium and the type of ligands attached to the central tellurium atom, (2-phenyl-azophenyl-C,N')tellurium(IV) trichloride, Te-N 2.417 Å[29, 103] , dimethyldithiocarbamato(2-phenylazophenyl-C,N')tellurium(II) , Te-N 2.340Å [29,104], acetato(2-phenylazophenyl-C,N') tellurium(II), Te-N 2.260 Å, (2-phenylazophenyl-C,N')thio cyanatotellurium(II), Te-N 2.243 Å [29,105] and (2-phenylazo -phenyl-C,N)tellurium(II) chloride, Te-N 2.230 Å[102]. The trend which seems to be indicated from this study, is that the tellurium-nitrogen interaction becomes shorter and hence stronger as the ligands attached to the tellurium become more electronegative and tellurium itself becomes a stronger Lewis acid.

The theme of this work was taken up and expanded by the work of N. I. Al-Salim, who investigated the synthesis and properties of some novel tellurium-containing ligands[30]. The primary interest in this study, was the synthesis of potentially multidentate ligands containing both tellurium and a donor atom such as nitrogen or oxygen. The single crystal study of the potentially polydentate tellurium ligand, 1,6-bis-2-butyltellurophenyl-2,5-diazahexa-1,5-diene reported in that investigation contains a Te.....N distance of 2.773 Å[30,106], which is well within the van der Waals distance (3.61 Å [107] or 3.7 Å[108] ). This Bis-telluride ligand was in turn reacted with

mercury chloride to form a one to one complex. In this complex, no mercury-nitrogen interaction was observed. The ligand was simply coordinated to the mercury through the two tellurium atoms, giving the environment around mercury as tetrahedral and leaving the tellurium-nitrogen interaction intact.

# CHAPTER TWO

# EXPERIMENTAL

## 2.1 Crystal Growth

Crystals were grown from a saturated solution of the material dissolved in a suitable spectro-grade solvent, (approximately 5 milligrams of material in 50 millilitres of solvent), placed in a constant temperature environment. As the solvent slowly evaporates the crystalline material is deposited from the saturated solution and crystal growth commences. Should the solution deposit only microcrystallites at the surface of evaporation, then the solvent evaporation process is too rapid for crystal growth to become established and needs to be slowed. Once the crystals have reached the desired proportions, ideally 0.2 to 0.3 $mm^3$, they are separated from the solvent medium by filtration and dried under vacuum. The sample should not be allowed to evaporate to dryness, as this will deposit any impurities present.

It may prove necessary to try a variety of solvents or adopt an alternative crystal growing technique such as mixed solvents, if a suitable crystalline specimen is not obtained initially. The production of suitable crystals for X-ray analysis is sometimes a tedious and difficult process, giving weight to the opinion that crystal growing is an art rather than a science.

## 2.2 Crystal Selection and Mounting

Under a Vickers polarising microscope, the extinction of the crystal is examined. If the crystal is single rather than "twinned" or "split" it should extinguish plane polarised light uniformly along its length four times when rotated through 360°. Failure to

do this results in the rejection of the crystal, and a selection of another.

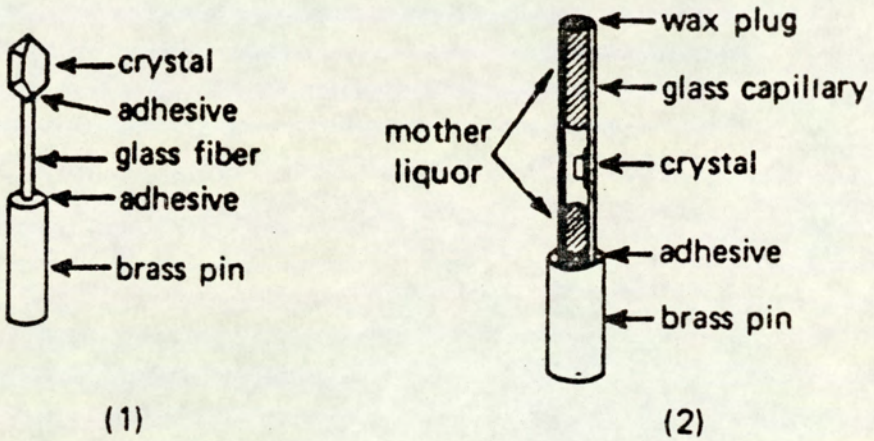
Once a crystal is selected, it is mounted on a glass fibre filament by a means of a contact adhesive or encapsulated in a capillary tube, if the crystal is unstable. (Fig.2.1). The capillary/glass filament is in turn attached to the goniometer head with either wax or plastacine (Fig. 2.2)[109].

## 2.3 X-Ray Analysis

### 2.3.1 Preliminary Photgraphic Analysis

Preliminary photographic analysis of the crystals took place on a Weissenberg camera (Fig2.3)[110], using $CuK_{\alpha}$ radiation. The crystal is set up optically, so that one of the crystal edges is perpendicular to the direction of the X-ray beam. A single oscillation photograph is produced initially, as the diffracted X-rays fall on the cylindrical film placed co-axially with the axis of rotation. As rotation proceeds, through about a twenty degree arc, various families of planes are brought in turn into an angular position such that the Bragg equation ($n\lambda = 2d\sin\theta$) is momentarily satisfied by the particular values of the spacing d and the glacing angle $\theta$ for the set of planes in question. A reflected beam is thus produced at a deviation of $2\theta$ on one side or other of the direct beam in a horizontal plane through the beam (Fig.2.4)[111]. The reflected beams are recorded on the film as a series of darkened spots. Each momentary appearance of a particular beam, as the crystal passes through the appropriate position will produce only a very slight darkening of the photographic emulsion. The camera

**Figure 2.1 Crystal Mounting Techniques**




The second technique is mainly used for mounting protein crystals.

**Figure 2.2 Gonimeter Head with Crystal Mounted**

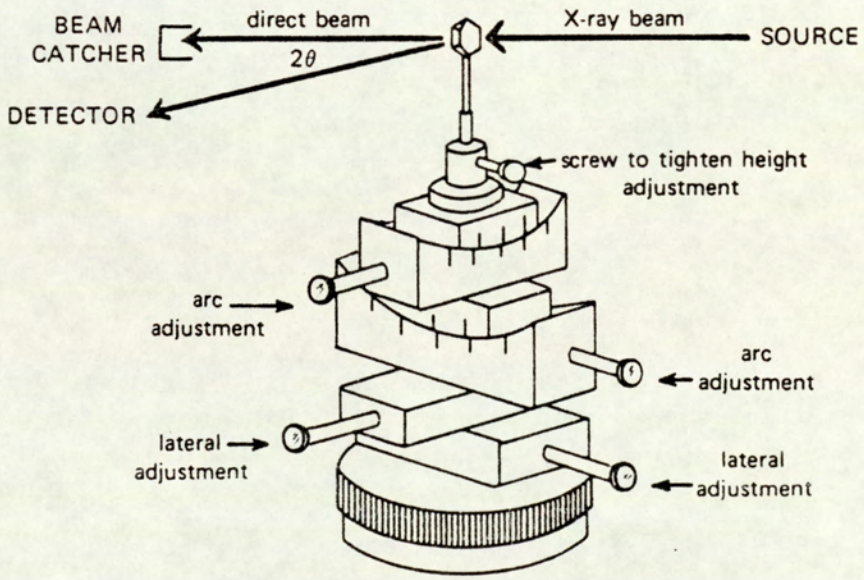
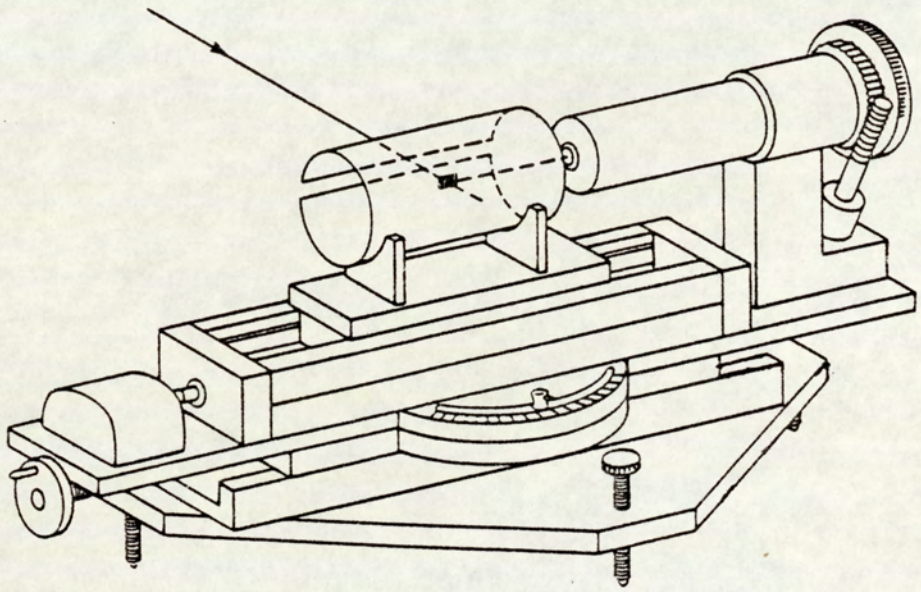


**Figure 2.3 Schematic View of a Weissenberg Camera**



**Description of Camera:**

The components consist of:

1) Goniometer head which carries adjusting arcs mounted at 90° to each other.

2) Telescope for assisting the setting of the crystal.

3) Mechanism for rotating or oscillating the crystal perpendicular to the beam.

4) Arrangement to hold a cylindrical camera cassette around the crystal.

5) Collimator to direct X-rays onto crystal.

6) Beam stop to catch the undeviated beam.

Figure 2.4 . Basic geometry of the Oscillation method showing how diffraction spots are recorded on a cylindrical film placed around the crystal

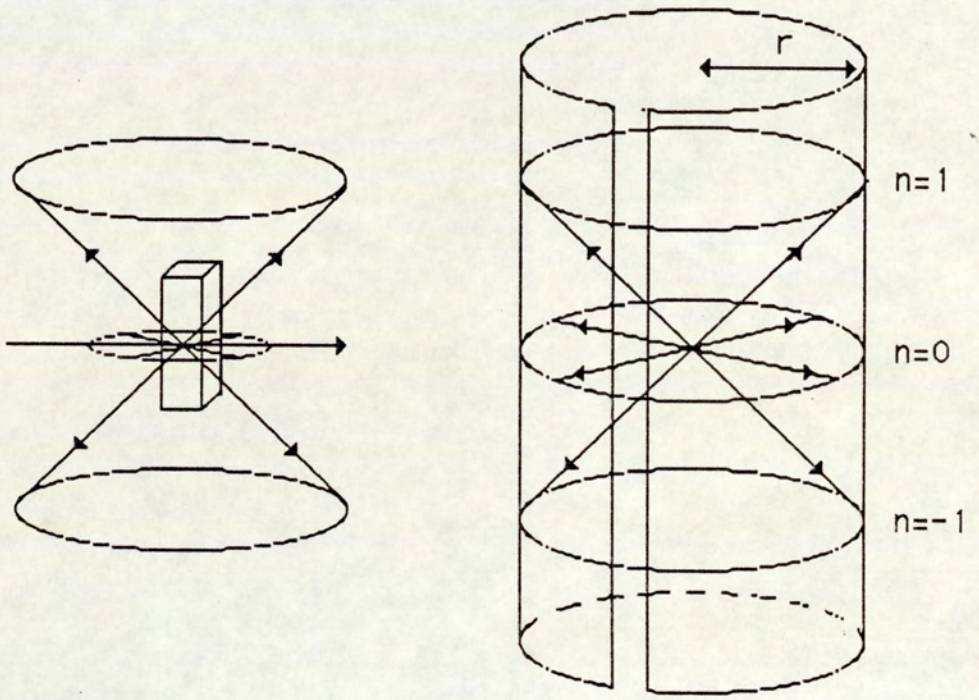
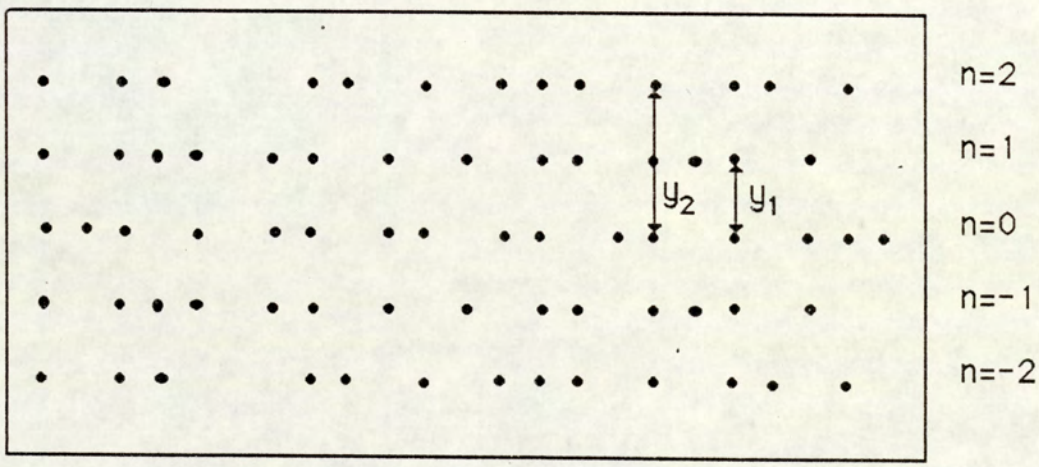



Figure 2.5 Sketch of a Single Oscillation Picture




For a known wavelength ($\lambda$) the following equation provides a convenient method for determining the unit cell spacings (a).

$$a = \frac{n\lambda}{\sin\{\tan^{-1}(y_n/r)\}}$$

r=radius of camera (mm)
y=distance of the $n^{th}$ layer line from the zero layer line.

is left to run for a period sufficiently long enough to produce a photograph of the required density, usually of the order of half an hour. The opened film, when developed consists of spots lying along straight lines called layer lines. (Fig.2.5)[111].

This preliminary investigation allows the crystallographer to examine whether infact the crystal is single and to assess how well the crystal diffracts. On examination, should the spots of the layer line be "split" rather than single dots, then the crystal is rejected.

The single oscillation photograph provides a means of measuring the length of the unit cell around which the crystal is oscillating (see fig.2.5 for calculation).

### 2.3.2 Detailed Photographic Analysis

In the event of a more in-depth photographic examination, the accurate orientation of the crystal is required. This is achieved by taking a series of single and double oscillation photographs[110]. It is possible to index the spots of the layer lines on rotation photographs, although it is not a simple task due to the possibility of overlapping reflections. This difficulty is overcome by moving film goniometers. The most widely used and the technique adopted for this work, is that due to Weissenberg.

As the crystal is rotating, the cylindrical film moves bodily along the axis of rotation, a complete back and forth motion takes place as the crystal is rotated through approximately 200° and back again. The gap between the two screens is adjusted to permit the passage of a selected cone of reflections (Fig.2.6). The

position of the slotted screens, having been predetermined by taking a zero layer line photograph. The motion of the film means that one layer of spots is spread over the whole film (Fig.2.7)[111].

The interpretation of the Weissenberg photograph, provides information about the cell axial lengths, angles and any systematic absences.

### 2.3.3 Automatic Counter Techniques

Photographic techniques for X-ray analysis of crystals, have largely been superseded by counter methods of recording intensities. Many different designs of diffractometer are used to facilitate the indexing of "reflections" and the measurement of intensities. The particular diffractometer used in this study was an Enraf-Nonius CAD-4 four circle diffractometer, which is a kappa geometry diffractometer.

The schematic set up of an Enraf-Nonius CAD-4 diffractometer is illustrated in Figure 2.8. The essential difference between this type of diffractometer and the "classical" Eurlerian four circle diffractometer, is that, instead of possessing a full CHI ($\chi$) circle, the Kappa geometry diffractometer possesses a Kappa arc which is inclined at 50° to the omega circle.

The unit is controlled by a Digital Equipment Corporation PDP-8/A computer. After positioning the crystal on the diffractometer, and before the data collection can be initiated the orientation matrix must be determined. This includes the unit cell

Figure 2.6. Sketch of the slotted screen arrangement when taking a Weissenberg photograph

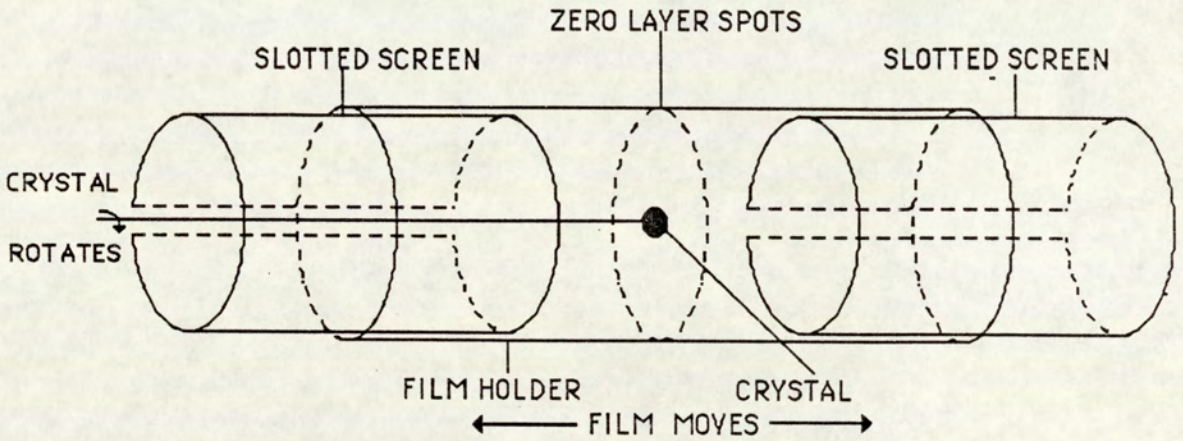
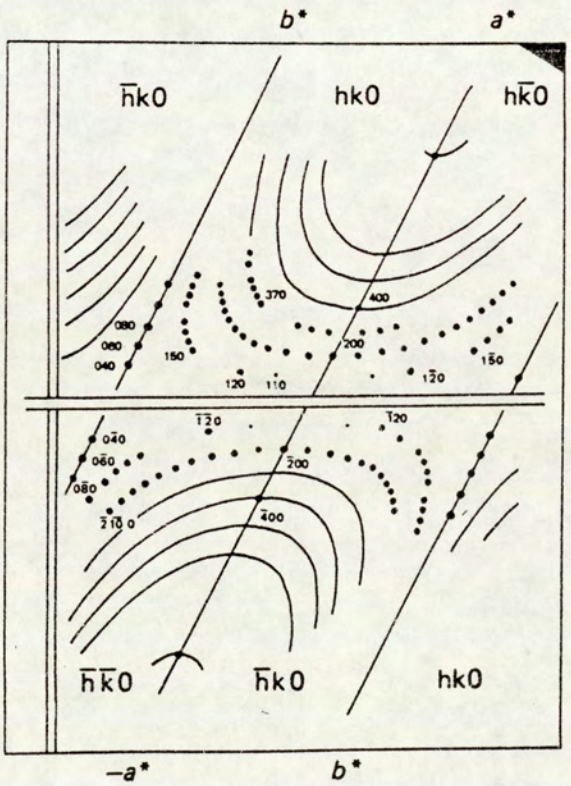



Figure 2.7. Sketch of a Weissenberg photograph, with the festoons partially drawn in




Equation (1)

$$\frac{X_{hkl}}{2\pi r} = \frac{4\theta_{hkl}}{2\pi}$$

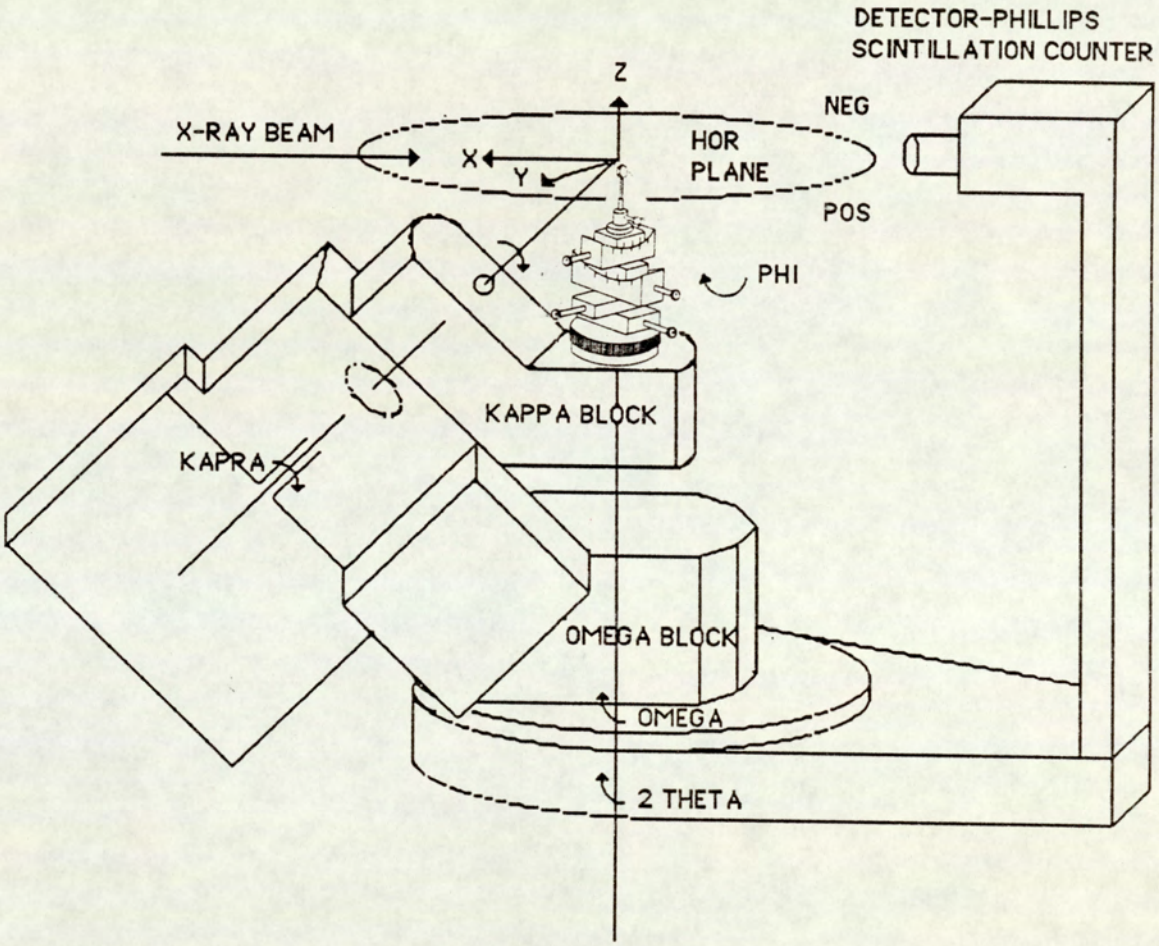
Equation (2)

$\lambda = 2d_{hkl}\sin\theta_{hkl}$

use $\theta_{hkl}$ found in equation (1) to find $d_{hkl}$ and inter axial angels from separation of the axial lines (110)

$X_{hkl}$ = distance (mm) between spots.
r = radius of camera (mm)
$\theta$ = diffraction angle.
$\lambda$ = wavelength (Å)

Figure 2.8 Enraf-Nonius CAD-4 Diffractometer.




(Graphite monochromated MoK$_\alpha$ radiation generated using an Enraf -Nonius generator run at 50KV and 20mA)

paramaters. The following routines are used to achieve this:

### 2.3.3.1 Search

The diffractometer is set in motion using this routine, as it provides a starting point, whereby the diffractometer searches through reciprocal space to locate and centre up to twenty-five reflections. The setting angles for these reflections are stored in the memory of the computer (REFDUMP), and form the basis for calculating the orientation matrix and the unit cell parameters.

The operator may vary the point in reciprocal space at which the search commences by re-setting the values of theta, chi and phi, fed into the computer. The speed of the phi motor and the discrimination factor for the peak to background ratio can also be controlled by the operator at this point.

### 2.3.3.2 Index

Using the reflecting positions stored in REFDUMP the INDEX routine produces an orientation matrix, the corresponding primitive cell and assigns indices to the stored reflections. The system executes a number of calculations in sequence. Initially, a collection of vectors ($\underline{V}$), in x,y,z, coordinates is composed, containing the scattering vectors ($\underline{R}_i$) derived from REFDUMP ($\leq$25), along with the sum and difference vectors, (Ri+j) of each combination of two scattering vectors ($\leq$600). From the collection of vectors, ($\underline{V}$), three vectors are drawn:

$\underline{R}_1$ = the shortest vector from ($\underline{V}$),

$\underline{R}_2$ = the shortest vector from ($\underline{V}$), which is the most perpendicular to $\underline{R}_1$,

$\underline{R}_3$ = the shortest vector from ($\underline{V}$), which is the most perpendicular to the plane through $\underline{R}_1$ and $\underline{R}_2$.

$\underline{R}_1$, $\underline{R}_2$ and $\underline{R}_3$ constitute the columns of the preliminary orientation matrix R(3,3). After the routine has initiated a series of internal checks successfully, the priminary indices are calculated thus,

$$R(3,3) * \begin{vmatrix} x_i \\ y_i \\ z_i \end{vmatrix} = \begin{vmatrix} h_i \\ k_i \\ l_i \end{vmatrix}$$

Indices calculated in this manner are most likely to be fractional, so they are multiplied by a common multiplication factor m, (which is determined by a least squares minimization procedure) and rounded off to integers.

The next cycle attempts to locate smaller base vectors, using the original vectors of R(3,3) as the starting set. Each vector of ($\underline{V}$), would be equal to $n_i R_1 + m_i R_2 + p_i R_3$ where $n_i$ $m_i$ and $p_i$ are integers, if the preliminary matrix is indeed correct. However, if the preliminary matrix is incorrect, there will be a

residual vector. In this instance, a series of two dimensional comparisons are applied to minimize the quantity, $V_1$-$n_i R_{1(2or3)}$. When a residual vector is more suitable to serve as a base vector, it replaces one of the original base vectors in the orientation matrix. A least squares procedure using the reflections in REFDUMP is applied to the new base vectors whilst checks are maintained to ensure the integrity of the Miller indices of the reflections. Ultimately, the refinement of the base vectors occurs using the reflections whose integrity has been preserved.

Index, by convention, works from a normalised primitive unit cell, such that A<B<C and the angles are all greater or less than ninety degrees. The Niggli matrix is also given. This can be used as an aid to determine whether or not the lattice is non-primitive, and so enables the operator to choose the most probable lattice.

To refine the unit cell and orientation matrix, the reflections are remeasured by the "SETANG" rountine, and then a "LEAST SQUARES" procedure is applied. Finally, "TRANS" tests the derived primitive cell for higher symmetry centred cells. Should the test confirm a higher symmetry centred cell, the routine provides the facility to transform the cell and re-index the reflections stored in REFDUMP.

#### 2.3.3.3 Data Collection

The $\omega/2\theta$ method of data collection, ie moving crystal, moving detector, was used in all cases of data collection. The data collection parameters were specified using the "DATCIN" programme. This programme provides a means to eliminate the

measurement of systematically absent reflections, allows the setting of the maximum and minimum range of θ values, controls the measurement of the intensity and orientation controls and facilitates the setting of the scan speed for a pre-scan of the intensities.

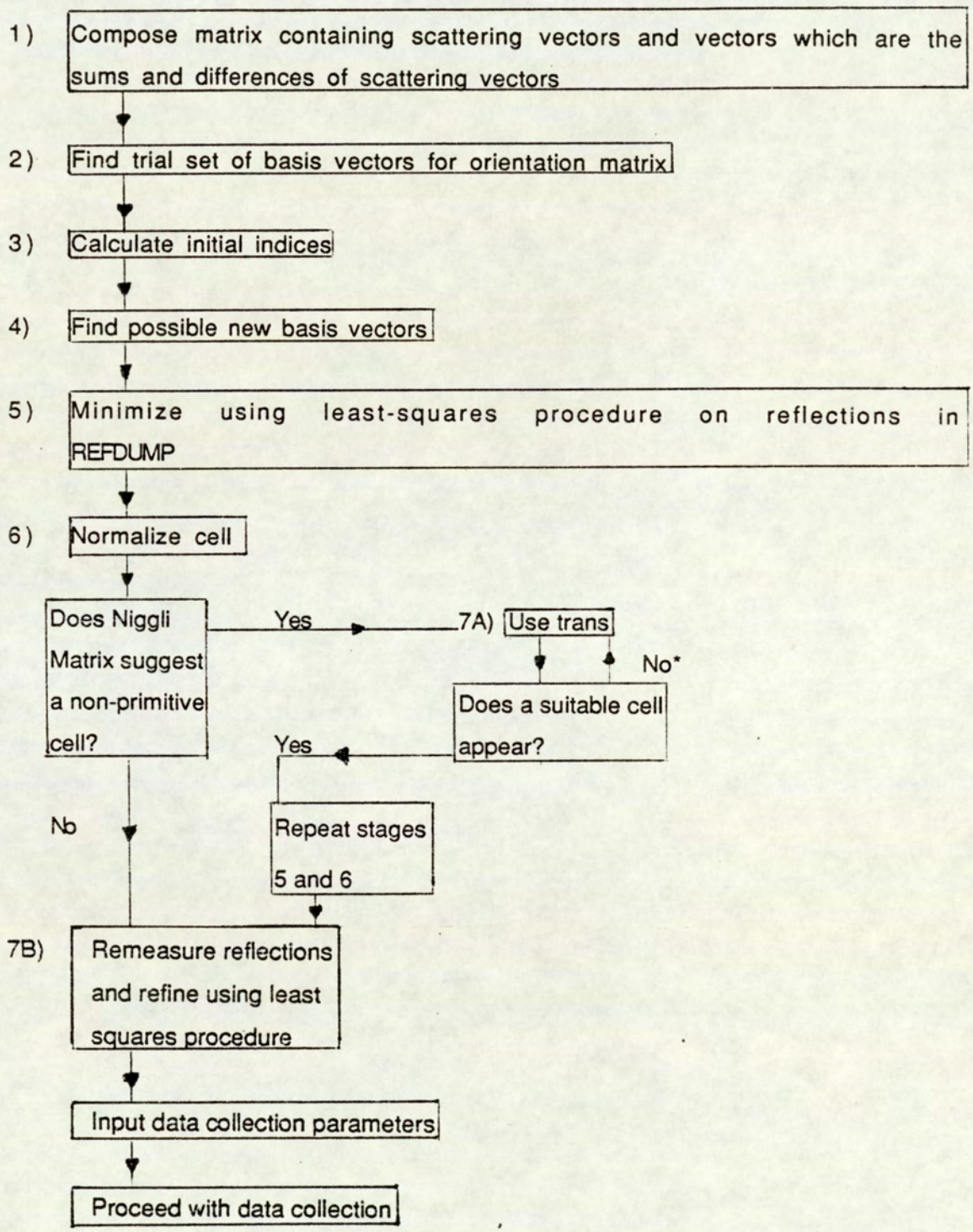
The measurement of intensities, at the specified pre-scan rate, will divide into three types. There will be those which are of acceptable quality such that the peak intensity is sufficiently greater than the background, secondly there will be peaks which are so weak not to warrant collection, as the peak intensity is roughly equivalent to the background and lastly there will be peaks which need collecting at a slower speed. In the last instance, the scan speed to be used will be calculated by the programme.

In all the instances in this study, the θ limits were set between 2 and 25°, apart from that is, the analysis of *p*-ethoxyphenyl[2-(2-pyridylphenyl]telluride where the range was 2 - 22.5°. Three intensity and orientation control reflections were used. During the data collection the standard reflections are remeasured at regular intervals. The orientation controls facilitate the recalculation of the orientation matrix, should this recalculation occur it may indicate the necessity to recentre the crystal in the X-ray beam. The intensity controls provide a means of monitoring any change in intensity, due for instance, to the deterioration of the crystal in the X-ray beam. If there is a change in the standard intensity values with time, a correction needs to be applied.

As the reflections are scanned the results are stored on magnetic disc, and simultaneously printed out for visual examination on a DECWRITER. At the completion of the data collection the operator may wish to refine or check the unit cell parameters, using higher values of h,k and l, taken from the print out. These high order reflections may be included into the list in REFDUMP, once room is made by deleting the requisite number of reflections to allow their inclusion. The reflections in the modified list are in turn remeasured using the SETANG routine and refined by the "LEAST SQUARES" procedure.

The results are transferred from the disc to magnetic tape at the end of the data collection. The visual print-out provides the operator with a convenient means of checking the data for systematic absences, should there be any. This information in conjunction with the type of Bravais Lattice, allows the assignment of a space group. This procedure can be summarized as illustrated by the following flow diagram, Fig. 2.9.

**FIGURE 2.9 A flow diagram illustrating the stages involved in collecting unit cell and intensity data using an Enraf-Nonius CAD-4 diffractometer.**




* If after several attempts no suitable cell appears, it is reasonable to assume that the crystal is defective in some way.

## 2.4 Computer Analysis

### 2.4.1 Reduction of Diffractometer Data

The raw intensity data was read from the magnetic tape into the Birmingham University Honeywell Multics Computer. The data was then reduced, using a programme called datred 4, provided by Dr T A Hamor. This programme provides the following facilities:

(i) Background subtraction: The general method for background subtraction is described in Stout and Jensen[110], this may be extended to counting times for peak and background as follows,

$$N_pK = N_T - r\,(N_{b1} + N_{b2})$$

where $N_T$ = total count, $N_pK$ = peak count, r = 1 and $N_{b1}$ and $N_{b2}$ are the background counts each collected over a period 1/r x peak count time.

(ii) Lorentz and polarization corrections (see introduction)

(iii) A simple linear intensity correction, based on the intensities of the standard reflections.

(iv) The programme provides the output in the form of h,k,l, $F_{obs}$ and $\sigma F_{obs}$, where $\sigma F_{obs}$ is calculated by the programme on the basis of counting statistics.

The standard deviation due to counting statistics is calculated by:

$$\sigma^2 pK = \sigma^2_T + \sigma^2_{b1} + \sigma^2_{b2}$$

where $\sigma^2_T = N_T$ and $\sigma_{b1} = r\sqrt{N_{b1}}$

The final standard deviation which includes an expression for variation in the main intensity beam can be expressed as follows:

$$\sigma^2_I = \sigma^2_p k + (f N_p k)^2$$

where f is a parameter which depends upon the constancy of the X-ray beam, in our case, a value of 0.01 was taken.

### 2.4.2 Crystallographic Calculations

The reduced data is now in a form suitable for use in integrated programmes which perform crystallographic calculations. The programmes used exclusively in this work were SHELX 86[11] and SHELX 76[12], both of which allow the solving of structures via the classical "heavy atom" or direct methods route. The SHELX system of programmes sets up complex neutral atomic scattering factors for all atoms used in this study [11,12] except for tellurium. The scattering factors used for this element were those defined by Cromer and Mann[112].

The programmes and route used in each structural solution were as follows; for the structural studies of dimethyldithio -carbamato-2-(2-quinolinyl)phenyltellurium(II) and (2-N,N-dimethylbenzylamine-C,N')tellurium(IV) tribromide, the direct methods option was employed using SHELX 86[11]. This facilitated the location of the tellurium and bromine atoms in the second structure, whilst in the first analysis the position of the tellurium and sulphur atoms in conjunction with the majority of the molecule were revealed. The heavy atom route was adopted

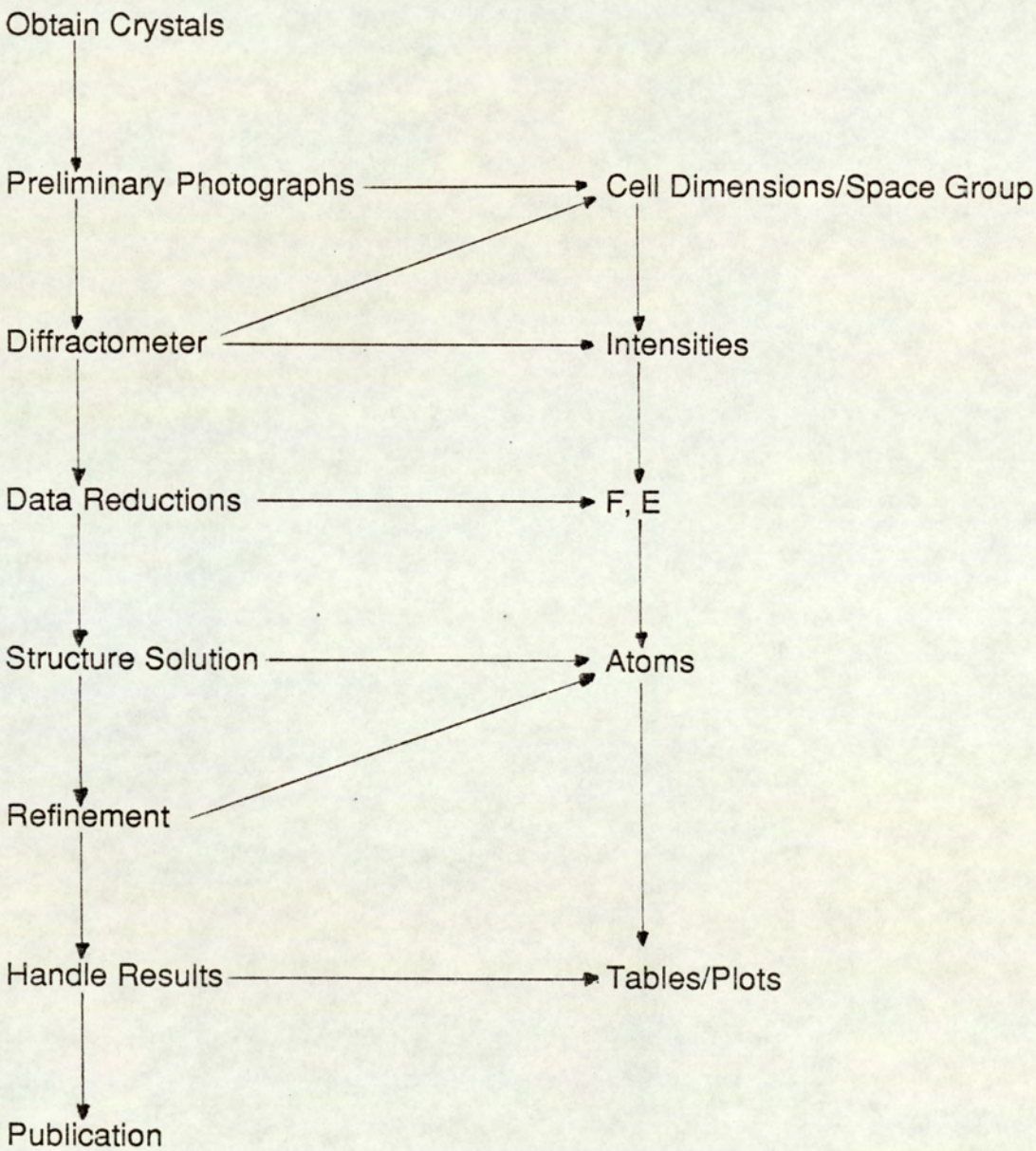
using SHELX 86[11] for the location of the tellurium atoms in 2-dichloro(butyl)tellurobenzaldehyde, bis[2-(N-hydroxy)imino-phenyl] ditelluride and 2-dichloro(butyl)telluro-N-dimethylbenzyl -ammonium chloride. In both of the chlorine containing structures not only were the tellurium positions revealed, but so were the positions of many of the lighter atoms. SHELX 76[12] was used in all other analyses. In the cases of bis[2-(2-pyridyl)phenyl]-tritelluride, dimethyldithiocarbamato[2-(2-pyridyl)phenyl]-tellurium(II) and *para*-ethoxyphenyl[2-(2-pyridyl)phenyl]telluride, the coordinates of the tellurium atoms were found using the heavy atom approach, where direct methods was used to find the tellurium position in 2-(2-pyridyl)phenyltellurium(IV) tribromide. In each of the crystal structures, the remaining non-hydrogen atoms were located by Fourier-difference synthesis, using SHELX 76[12]. The hydrogen atoms in each structure were placed in calculated positions (C-H 1.08Å) and allowed to "ride" on their respective carbon atoms in the subsequent least-squares refinements, unless they were able to be located from a difference map. Empirical absorption corrections[113] were applied in the cases of 2-(2-pyridyl)phenyltellurium(IV) tribromide, (range of transmission factors 0.524-1.083) and (2-N,N-dimethylbenzylamine-C,N')tellurium(IV) tribromide (range of transmission factors 0.145-3.984)[113].

Mean plane calculations and torsion angles were calculated using Mpl86[114], on the University of Birmingham Honeywell Multics system. PLUTO 78[115] at the University of Manchester Regional Computer Centre (UMRCC) was used for all molecular

plotting in this work. The programme allows the plotting of either unit cell contents or individual molecules, with the options for projections, prespective views or stereo representation. The output can be drawn either as a ball and stick representation or as a space filling diagram. A number of orientation options are permitted, with the default option producing the minimum overlap orientation.

The determination of crystallographic structures from initiation to completion can be summarised as illustrated in the following flow diagram, Fig. 2.10.

**Figure 2.10 A flow diagram illustrating the stages in the determination of a crystal structure from initiation to completion**



# CHAPTER THREE

# RESULTS AND DISCUSSION ON INDIVIDUAL STRUCTURES

## 3.1 Polytelluride Structures

### 3.1.1 Bis[2-(2-pyridyl)phenyl]tritelluride, (1)

At the initiation of this study, a number of examples of the tritelluride ion $[Te_3]^{2-}$ had been recorded[116-118], but no evidence could be located as to the existence of a non-ionic tritelluride single crystal study. It was anticipated that this structural elucidation would provide the detailed structural evidence of the first non-ionic tritelluride. During this investigation however, the structure of bis-(tris(trimethyl silyl)methyl)tritelluride was reported[119]. Even though there are significant differences between the two structures, this analysis provided the first reported example of a non-ionic tritelluride.

The deep blood red crystals were supplied for crystallographic examination by N Al-Salim of this department. The crystal chosen for the investigation was plate-like, measuring 0.75 x 0.47 x 0.05 mm.

**Crystal Data**

$C_{22}H_{16}N_2Te_3$, Mr = 691.2, monoclinic space group I2/C, a = 14.721(3) Å, b = 9.290(4) Å, c = 15.996(10) Å, ß = 106.39(3) Å, U = 2098.7 $Å^3$, Z = 4, $D_c$ = 2.188 $gcm^{-3}$, F(000) = 1272, $\lambda$(Mo-$K_\alpha$) = 0.71069 Å, $\mu$ = 4.31 $mm^{-1}$.

1639 unique reflections in the range $2<\theta<25$ with $I>2.5\theta(I)$ were used in the structural analysis. Least squares refinement

was terminated when all the shifts were less than $0.001\sigma$ and R and Rw were 0.064 and 0.088 respectively, $W=1/(\sigma^2(F)+0.005F^2)$.

Final atomic coordinates for bis[2-(2-pyridyl)phenyl] tritelluride are given in table 3.1, with bond distances, bond angles, torsion angles and mean plane calculations given in tables 3.2 and 3.3 respectively. A view of the molecule and a stereoscopic packing diagram are shown in figures 3.1 and 3.2.

The asymmetric unit consists of half of the molecule, as the molecule exhibits two fold symmetry with the central tellurium atom lying on a crystallographic two fold axis. The coordination about each terminal tellurium can be considered as essentially trigonal bipyramidal, with the two lone pairs of electrons on tellurium and the carbon of the phenyl ring equatorial and the central tellurium atom with the pyridyl nitrogen axial. Thus the position *trans* to the Te-C bond is unoccupied and each half of the molecule can be considered as "T-shaped"[40].

The Te(1)-C(1) distance [2.130(9) Å] is in reasonable agreement with the sum of the Pauling single bond covalent radii for tellurium 1.37 Å and a $sp^2$ hybridised carbon 0.74 Å[108], and with the analogous bond in ($C_7H_5OTeBr$) 2.08 Å[120], $C_7H_6NTe$ 2.08(1)-2.09(1) Å[121], $C_{18}H_{18}O_4SeTe_2$ 2.123(8) Å[122], and $C_{14}H_{14}O_2SeTe_2$ 2.093(17), 2.106(18) Å[123]. The bond lengths and angles in the phenyl ring are unexceptional, and are detailed in table 3.2. The ring itself is essentially planar, with a route mean squared deviation of 0.009Å.

The Te(1)-N(1) distance, 2.554(7) Å is noticeably longer than

the sum of the covalent radii [2.07 Å[108] or 2.24 Å for axial Te-N[40,108]] but is considerably shorter than the sum of the van der Waals radii. [3.70 Å[108], 3.61 Å[107]]. This secondary interaction[39] although longer than similar Te-N contacts[102,104,105] is analogous to that identified in other trichalcogens where an oxygen is acting as the donating atom in the system, forming a comparable five membered chelate ring[122,124]. The Te(1)-N(1) interaction is of additonal note, as it appears to hold the 2-(2-pyridyl)phenyl ligand in an almost planar geometry with only a slight bending of one ring relative to the other, forming a shallow V shape (Table 3.3). Bis[2-(2-pyridyl)phenyl]tritelluride appears to owe its stability to the Te-N interaction, whereas the bulky $(Me_3Si)_3C$- groups are believed to contribute to the stability of Bis(tris(trimethylsilyl) methyl)tritelluride[119].

The Te-Te bond length [2.776(1) Å] is in good agreement with the sum of the Pauling covalent single bond radii[108] and with typical values identified for Te-Te bonds in analogous complexes eg bis(tris(trimethylsilyl)methyl)tritelluride[119], di-potassium tritelluride[117], barium tritelluride[118], diphenyl ditelluride [125] and *p*,*p*-dimethoxydiphenyl ditelluride[125] which lie in the range 2.710 - 2.805 Å. The terminal telluriums related by the molecular two fold symmetry lie 4.265 Å apart with a Te(1)-Te(2)-Te(1) angle of 100.4(2)°.

There appear to be no significant intermolecular contacts less than the sum of the involved atoms van der Waals radii. The closest Te-Te intermolecular contact is 4.678 Å, which is well

beyond the sum of the van der Waals radii for tellurium[107,108]. An interesting feature in the crystal is the parallel stacking of the organic ligands.

Table 3.1 . Fractional atomic coordinates (x10$^5$ for tellurium and x10$^4$ for all other non-hydrogen atoms) with e.s.d.'s in parentheses for Bis[2-(2-pyridyl)phenyl]tritelluride.

| | x | y | z |
|---|---|---|---|
| Te(1) | 7063( 4) | 50965( 5) | 15050( 3) |
| Te(2) | 0( 0) | 31844( 8) | 25000( 0) |
| N(1) | 1619( 5) | 6771( 8) | 765( 5) |
| C(1) | 2154( 6) | 5107( 8) | 2254( 6) |
| C(2) | 2453( 6) | 4235( 9) | 3021( 5) |
| C(3) | 3403( 7) | 4236(10) | 3511( 6) |
| C(4) | 4033( 8) | 5141(11) | 3253( 9) |
| C(5) | 3747( 6) | 5970(10) | 2531( 6) |
| C(6) | 2826( 5) | 5967( 8) | 2019( 5) |
| C(7) | 2536( 7) | 6856( 8) | 1230( 6) |
| C(8) | 3146( 8) | 7702(12) | 937( 8) |
| C(9) | 2791(11) | 8492(12) | 155( 9) |
| C(10) | 1856(11) | 8344(12) | -297( 8) |
| C(11) | 1302( 8) | 7486(10) | 41( 6) |

Table 3.2. Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses for Bis[2-(2-pyridyl)phenyl]tritelluride.

| | | | | | |
|---|---|---|---|---|---|
| Te(1) | ---Te(2) | 2.776 ( 1) | C(6) | ---C(1) | 1.403 (11) |
| Te(1) | ---C(1) | 2.130 ( 9) | C(6) | ---C(7) | 1.467 (13) |
| Te(1) | ---N(1) | 2.554 ( 7) | C(8) | ---C(9) | 1.416 (18) |
| C(2) | ---C(3) | 1.397 (13) | C(8) | ---C(7) | 1.371 (12) |
| C(2) | ---C(1) | 1.431 (12) | C(9) | ---C(10) | 1.369 (21) |
| C(3) | ---C(4) | 1.396 (15) | C(10) | ---C(11) | 1.357 (15) |
| C(4) | ---C(5) | 1.352 (16) | C(11) | ---N(1) | 1.302 (12) |
| C(5) | ---C(6) | 1.373 (11) | N(1) | ---C(7) | 1.348 (13) |

| | | | | | |
|---|---|---|---|---|---|
| Te(2) | -Te(1) -C(1) | 99.1 ( 2) | Te(1) | -C(1) -C(6) | 121.4 ( 6) |
| Te(2) | -Te(1) -N(1) | 170.7 ( 2) | C(2) | -C(1) -C(6) | 118.8 ( 8) |
| C(1) | -Te(1) -N(1) | 71.6 ( 3) | C(9) | -C(8) -C(7) | 119.0 (11) |
| Te(1) | -Te(2) -Te(1) | 100.4 ( 2) | C(8) | -C(9) -C(10) | 118.7 ( 9) |
| C(3) | -C(2) -C(1) | 119.8 ( 9) | C(9) | -C(10) -C(11) | 118.4 (11) |
| C(2) | -C(3) -C(4) | 118.5 ( 9) | C(10) | -C(11) -N(1) | 123.3 (11) |
| C(3) | -C(4) -C(5) | 121.5 (10) | Te(1) | -N(1) -C(11) | 128.3 ( 7) |
| C(4) | -C(5) -C(6) | 121.7 ( 9) | Te(1) | -N(1) -C(7) | 110.9 ( 5) |
| C(5) | -C(6) -C(1) | 119.5 ( 8) | C(11) | -N(1) -C(7) | 120.8 ( 8) |
| C(5) | -C(6) -C(7) | 120.8 ( 7) | C(6) | -C(7) -C(8) | 123.9 ( 9) |
| C(1) | -C(6) -C(7) | 119.7 ( 7) | C(6) | -C(7) -N(1) | 116.4 ( 7) |
| Te(1) | -C(1) -C(2) | 119.8 ( 6) | C(8) | -C(7) -N(1) | 119.7 ( 9) |

**TABLE 3.3**

**Mean Plane Analysis for Bis[2-(2-pyridyl)phenyl] tritelluride.**

Phenyl Ring - Perpendicular Distance to Plane.

| | |
|---|---|
| 0.00160 | C1 |
| 0.01026 | C2 |
| -0.01432 | C3 |
| 0.00638 | C4 |
| 0.00586 | C5 |
| -0.00978 | C6 |

Mean Dev = 0.008034 R.M.S Dev = 0.008978

Pyridyl Ring - Perpendicular Distance to Plane.

| | |
|---|---|
| 0.00275 | N1 |
| 0.00016 | C7 |
| -0.00727 | C8 |
| 0.01165 | C9 |
| -0.00914 | C10 |
| 0.00185 | C11 |

Mean Dev= 0.005471 R.M.S Dev= 0.006871

Angle Between Planes 2.81(2)°.

Torsion Angles

| | |
|---|---|
| N(1)-C(7)-C(6)-C(5) | 179.4 |
| N(1)-C(7)-C(6)-C(1) | -0.7 |
| C(8)-C(7)-C(6)-C(5) | 1.8 |
| C(8)-C(7)-C(6)-C(1) | -178.4 |
| C(1)-Te(1)-Te(2)-Te(3) | 97.3 |

Figure 3.1. The structure of Bis[2-(2-pyridyl)phenyl]tritelluride showing the atom numbering.

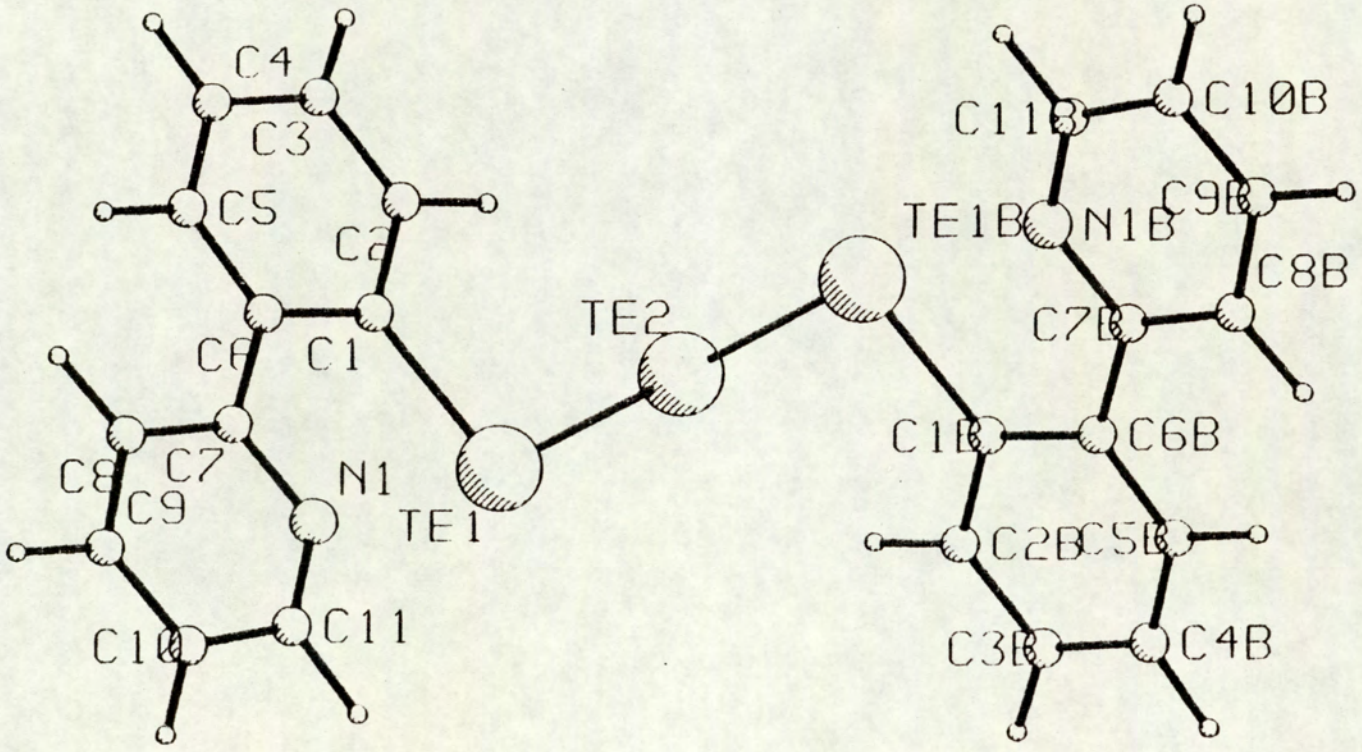
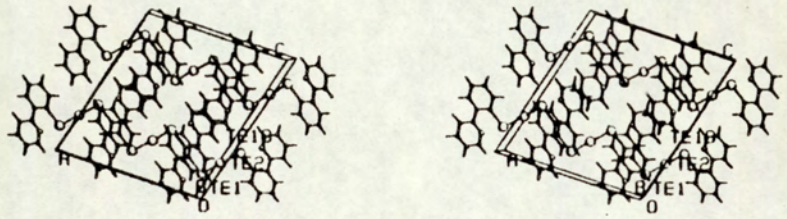


Figure 3.2. Stereoscopic packing diagram of Bis[2-(2-pyridyl)phenyl]tritelluride.



### 3.1.2 Bis [2-(N-hydroxy)iminophenyl] Ditelluride, (2)

The synthetic route and chemical properties of this compound have been reported previously[33]. Originally it was anticipated that this synthetic pathway would yield the first cyclic ditelluride or a polymeric ditelluride. The crystal structure detemination undertaken was to unequivocally identify the product formed.

Brown crystals of the reaction product were grown from benzene by K Y Abid of this department. The dimensions of the crystal chosen for analysis were 0.475 x 0.625 x 0.300 mm.

Crystallographic analysis revealed that neither of the two envisaged products had been formed. The composition of the majority of the unexpected product was revealed by the combination of X-ray, Infrared and CHN results, however there was still some ambiguity as to the precise nature of the terminal group of the phenyl ring side chain. Initially the group was presumed to be $CH_3$, as this assignment tallied the theoretical and experimental C and H results[33], within analytical error. The structural refinement continued normally, all be it with slightly high thermal parameters for the two terminal methyl carbons. Assigning the terminal group of the phenyl ring as $CH_3$ did not completely explain the I.R. trace, so it was decided to refine the site occupancy factor for both peaks in the following least-squares cycle. Freeing this factor revealed an occupation of 1.56 and 1.59, for the two peaks designated as methyl carbons. This information lead to a rethink and a reassignment of the terminal groups from $CH_3$ to $NH_2$. Labelling the group $NH_2$ failed to

equate the theoretical and analytical nitrogen values and only succeeded in bringing the site occupancy factors down to 1.25 and 1.28. Clearly another rethink was required. Considering the terminal groups to be OH gave satisfactory site occupancy factors of 1.02 and 1.03 for each peak. On closer examination of the difference map, only one peak which could be attributed to hydrogen could be located in the vicinity of each oxygen. This additional evidence in conjunction with the CHN results and IR trace seem to confirm the terminal group in the phenyl ring side chain as OH.

**Crystal Data**

$C_{14}H_{12}N_2O_2Te_2$, Mr = 495.5, monoclinic space group $P2_1/n$ a = 15.790(3) Å, b = 6.340(8) Å, c = 15.903(6) Å, ß = 99.50(2)°, U = 1570.2 $Å^3$, Z = 4, $D_c$ = 2.096 $gcm^{-3}$, F(000) = 920, $\lambda$(Mo-$K_\alpha$) = 0.71069 Å, $\mu$ = 3.856 $mm^{-1}$.

The total number of reflections scanned in the range 2-25θ was 5668, with 2491 unique reflections with $I>2.5\theta(I)$ used in the structural analysis. The analysis was halted when all shifts were less than 0.01σ and R and Rw were 7.53% and 9.06% respectively; $w=1/[\sigma^2(F)+0.002F^2]$.

Final atomic coordinates for Bis[2(N-hydroxyl)iminophenyl] ditelluride are given in table 3.4, with bond distances, bond angles and mean plane calculations given in tables 3.5 and 3.6 respectively. A view of the molecule and a stereoscopic packing

diagram are shown in figures 3.3 and 3.4.

The distances Te(1) - C(1), 2.136(8) Å and Te(2)-C(8), 2.142(8) Å, are slightly longer than the sum of the Pauling single bond covalent radii for a tellurium-$sp^2$ carbon single bond, (2.11 Å)[108], but they are similar to values found in other diorganyl ditellurides, range 2.08-2.16 Å[125-128] and with the analogous bond in our tritelluride, 2.130(9) Å. The carbon carbon lengths and angles within the phenyl ring are analogous to standard values and are given in detail in Table 3.5. Interestingly, within the crystal there is parallel stacking of the phenyl rings, but these rings do not directly overlie one another within the stack.

The Te-N distances, 2.822(5) and 2.876(5) Å, are much greater than the sum of the covalent radii[108], but are within the van der Waals distance, 3.61 Å[107], and therefore maybe considered as a weak secondary interaction[39]. These distances are shorter than the recently reported Te-N interactions in $Me_2Te(OC_6H_4{=}CHC_6H_4NO_2)_2$, [2.962, 2.923 Å][129] where the oxidation state for tellurium is IV as opposed to a formal oxidation of I in this ditelluride. The aldoxime groups are planar and are orientated at 11.2(3)°, 4.1(2)° to the phenyl ring to which they are attached. The bond lengths and angles of the aldoxime groups are in reasonable agreement with previously published results[130].

The Te-Te bond length of 2.746(1) Å, is in good agreement with the sum of the Pauling single bond covalent radii (2.74 Å)[108] and can be compared to values in the crystal structures of a number of analogous compounds, range 2.697-2.715 Å[125-128].

The environment around each tellurium may be loosely described as trigonal bipyramidal, if the Te-N distance is considered significant and included in the coordination sphere. The molecule appears to twist about the Te-Te bond such that the planes of the ligands are approximately at right angles to each other.

Examination of packing distances shows that the shortest distances between tellurium atoms are between Te(1) and Te(1) (-x,-y,-z), 3.777(4) Å and between Te(2) and Te(2) (-x,-y,-z), 4.041(4) Å. These contacts are tending towards the van der Waals distance of 4.12 Å or 4.40 Å[107,108], and may thus represent only a very weak interaction between the molecules.

Table 3.4. Fractional atomic coordinates ($x10^5$ for tellurium and $x10^4$ for all other non-hydrogen atoms) with e.s.d.'s in parentheses for Bis-[2-(N-hydroxy)iminophenyl] Ditelluride.

| | x | y | z |
|---|---|---|---|
| Te(1) | -5300( 3) | 5060( 9) | 9620( 3) |
| Te(2) | -12014( 3) | 39820( 9) | 588( 3) |
| O(1) | 678( 6) | -4324(11) | 2173( 5) |
| O(2) | -1901( 7) | 8600(15) | -1650( 7) |
| N(1) | 106( 5) | -2604(11) | 2173( 5) |
| N(2) | -2090( 5) | 6804(13) | -1204( 5) |
| C(1) | -1023( 5) | 959(12) | 2122( 5) |
| C(2) | -1523( 5) | 2726(13) | 2238( 5) |
| C(3) | -1874( 5) | 2936(17) | 2969( 6) |
| C(4) | -1777( 6) | 1406(15) | 3580( 5) |
| C(5) | -1280( 6) | -313(15) | 3474( 6) |
| C(6) | -900( 5) | -620(13) | 2773( 5) |
| C(7) | -374( 6) | -2452(13) | 2724( 6) |
| C(8) | -2499( 5) | 2954(12) | -328( 4) |
| C(9) | -2794( 6) | 1147(14) | -2( 6) |
| C(10) | -3661( 6) | 501(19) | -199( 6) |
| C(11) | -4230( 6) | 1837(19) | -718( 7) |
| C(12) | -3929( 6) | 3552(20) | -1064( 7) |
| C(13) | -3077( 5) | 4206(15) | -868( 5) |
| C(14) | -2845( 6) | 6136(15) | -1300( 6) |

Table 3.5. Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses for Bis-[2-(N-hydroxy)iminophenyl] Ditelluride.

| | | | |
|---|---|---|---|
| Te(1) ---Te(2) | 2.746 ( 1) | C(4) ---C(5) | 1.370 (14) |
| Te(1) ---C(1) | 2.136 ( 8) | C(5) ---C(6) | 1.364 (13) |
| Te(2) ---C(8) | 2.142 ( 8) | C(6) ---C(7) | 1.438 (12) |
| O(1) ---N(1) | 1.415 (10) | C(8) ---C(9) | 1.370 (12) |
| O(2) ---N(2) | 1.399 (12) | C(8) ---C(13) | 1.393 (11) |
| N(1) ---C(7) | 1.253 (12) | C(9) ---C(10) | 1.414 (12) |
| N(2) ---C(14) | 1.252 (12) | C(10) ---C(11) | 1.401 (16) |
| C(1) ---C(2) | 1.400 (11) | C(11) ---C(12) | 1.340 (16) |
| C(1) ---C(6) | 1.431 (10) | C(12) ---C(13) | 1.393 (13) |
| C(2) ---C(3) | 1.375 (12) | C(13) ---C(14) | 1.478 (13) |
| C(3) ---C(4) | 1.363 (13) | | |

| | | | |
|---|---|---|---|
| Te(2) -Te(1) -C(1) | 100.3 ( 2) | C(5) -C(6) -C(7) | 119.7 ( 8) |
| Te(1) -Te(2) -C(8) | 100.0 ( 2) | N(1) -C(7) -C(6) | 121.4 ( 8) |
| O(1) -N(1) -C(7) | 121.2 ( 8) | Te(2) -C(8) -C(9) | 120.9 ( 6) |
| O(2) -N(2) -C(14) | 119.4 ( 8) | Te(2) -C(8) -C(13) | 120.1 ( 6) |
| Te(1) -C(1) -C(2) | 121.0 ( 5) | C(9) -C(8) -C(13) | 118.8 ( 8) |
| Te(1) -C(1) -C(6) | 120.4 ( 6) | C(8) -C(9) -C(10) | 122.4 ( 9) |
| C(2) -C(1) -C(6) | 118.5 ( 7) | C(9) -C(10) -C(11) | 117.3 (10) |
| C(1) -C(2) -C(3) | 120.3 ( 8) | C(10) -C(11) -C(12) | 119.7 ( 9) |
| C(2) -C(3) -C(4) | 121.4 ( 9) | C(11) -C(12) -C(13) | 123.0 (10) |
| C(3) -C(4) -C(5) | 118.5 ( 8) | C(8) -C(13) -C(12) | 118.5 ( 9) |
| C(4) -C(5) -C(6) | 123.5 ( 8) | C(8) -C(13) -C(14) | 124.9 ( 8) |
| C(1) -C(6) -C(5) | 117.8 ( 8) | C(12) -C(13) -C(14) | 116.5 ( 8) |
| C(1) -C(6) -C(7) | 122.5 ( 8) | N(2) -C(14) -C(13) | 121.8 ( 8) |

TABLE 3.6

**Mean Plane Analysis for Bis[2-(N-hydroxy)iminophenyl] Ditelluride.**

Phenyl Ring attached to Te1.

| Perpendicular Distance to Plane | Atom |
|---|---|
| -0.00231 | C1 |
| -0.00624 | C2 |
| 0.01409 | C3 |
| -0.01314 | C4 |
| 0.00449 | C5 |
| 0.00311 | C6 |

Mean Dev = 0.007228 R.M.S. Dev = 0.008613

Phenyl Ring attached to Te2

| Perpendicular Distance to Plane | Atom |
|---|---|
| 0.00652 | C8 |
| 0.00100 | C9 |
| -0.01887 | C10 |
| 0.03063 | C11 |
| -0.02356 | C12 |
| 0.00427 | C13 |

Mean Dev = 0.14142 R.M.S.Dev. = 0.017846.

Figure 3.3. The structure of Bis-[2-(N-hydroxy)iminophenyl] Ditelluride showing the atom numbering.

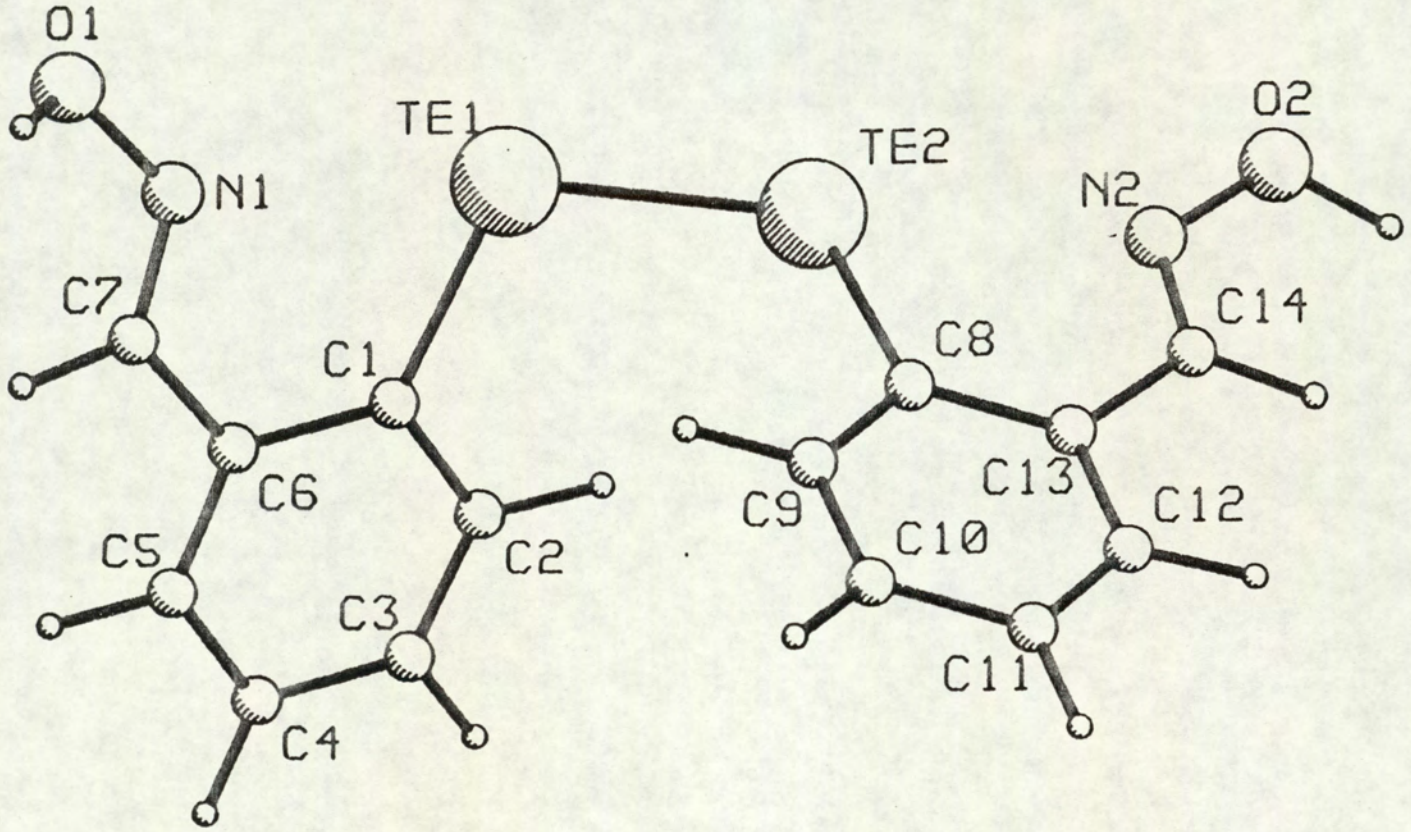
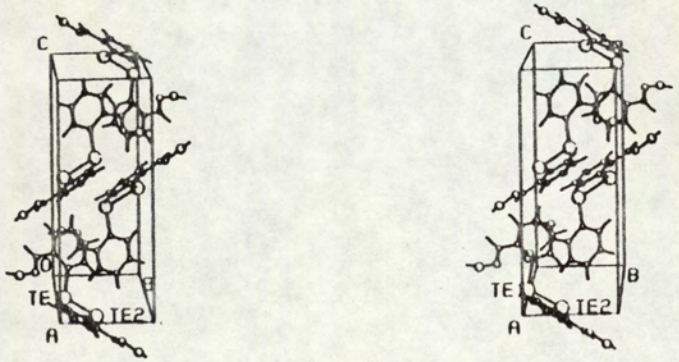


Figure 3.4. Stereoscopic packing diagram of Bis[2-(hydroxy)iminophenyl] Ditelluride.





## 3.2 Tellurium(IV) Structures

### 2-(2-Pyridyl)phenyltellurium (IV) tribromide, (3)

Analysis of previous single crystal studies of $RTeX_3$ compounds reveals a tendency for the molecules to link into either polymers (in the case of trichlorides)[87,131-133] or dimers (in the case of tribromides and triiodides)[133,134] via quite strong halogen bridge bonds. This phenomenon is absent when the organic group present is either bulky or contains a donor group. In such structures the molecules are either associated into weak dimers or are essentially monomeric[135-140].

The study of 2-(2-pyridyl)phenyltellurium(IV) tribromide provided an opportunity to examine an $RTeX_3$ compound with an electron donating nitrogen incorporated into an arguably non-bulky organic ligand. Interest in this study therefore naturally centred on whether this type of donor atom would facilitate the means of satisfying the Lewis acidity of the system by intramolecular complex formation and so prevent the formation of strong intermolecular halogen bridges. Apart from this aspect of the study, added interest came from the fact that 2-(2-pyridyl) -phenyltellurium(IV) tribromide formed a precursor stage in the preparation of both bis[2-(2-pyridyl)phenyl]tritelluride and dimethyldithiocarbamato[2-(2-pyridyl)phenyl]tellurium(II).

Bright yellow crystals of 2-(2-pyridyl)phenyltellurium(IV) tribromide were grown from acetonitrile by N Al-Salim of this department. The size of the crystal chosen for analysis was 0.40 x 0.20 x 0.10 mm.

**Crystal Data**

$C_{11}H_8Br_3NTe$, Mr = 521.5, triclinic space group. $P\bar{1}$ , a = 6.953(6) Å, b = 8.382(1) Å, c = 12.133(2) Å, $\alpha$ = 78.68(1)°, ß= 82.87(4)°, $\gamma$ = 87.14(4)°, U = 687.8 $Å^3$, Z = 2, $D_c$ = 2.519 $gcm^{-3}$, F(000) = 476, $\lambda$(Mo-$K_\alpha$) = 0.71069 Å, $\mu$ = 11.45 $mm^{-1}$.

The structural analysis was based on 1963 significant reflections [$I>2.5\theta(I)$] drawn from the original 2697 unique reflections scanned in the prescribed $\theta$ range. Least squares analysis was terminated when all shifts were less than 0.15$\sigma$ and R and Rw were 7.35% and 10.85% respectively. No weighting scheme was applied to the reflections.

Final atomic coordinates for 2-(2-pyridyl) phenyltellurium(IV) tribromide are given in table 3.7, with bond distances, torsion angles and mean plane calculations given in tables 3.8 and 3.9 respectively. A view of the molecule and a stereoscopic packing diagram are shown in figures 3.5 and 3.6.

The coordination about tellurium can be considered as essentially octahedral. In this arrangement the tellurium atom rests in the centre of the equatorial plane of the octahedron, which is defined by the pyridyl nitrogen, the phenyl ring carbon, [C(1)] and the bromine atom [Br(3)]. The fourth equatorial site may be considered to be either vacant or occupied by the lone pair of electrons on tellurium, if these are considered to be stereochemically active. The octahedron around tellurium is completed by two bromine atoms which occupy the axial sites

situated above and below the equatorial plane.

In an ideal octahedron the angles around the central atom are either 90° or 180°. In this structure the slight deviations from idealarity can be attributed to either constraints arising from the five membered chelate ring or to the fact that a lone pair of electrons occupies the fourth equatorial position between N(1) and Br(3). In particular the restricted N(1)-Te-C(1) angle at 78.2(7)° seems to be a consequence of the first factor and possibly so is the angle between N(1)-Te-Br(3) at 170.4(4)°. The latter angle is presumably also affected by the lone pair of electrons situated between N(1) and Br(3). In the axial direction the atoms are noticeably displaced from the lone pair of electrons which reduces the Br(1)-Te-Br(2) angle to 172.4(1)°. This type of distortion is similar to that observed in (2-phenylazophenyl-C,N')tellurium(IV) trichloride, where analogous angles were constrained in a like manner[103].

Within the structure, tellurium forms a single covalent bond with an $sp^2$ hybridised carbon atom. This distance [Te-C(1) 2.110(19) Å] although unremarkable in its own right, lies neatly within a range of values reported in previous structures for the same bond, range 2.11-2.14 Å [103,135,141,142]. The interaction between tellurium and nitrogen [2.244(14) Å] is considerably longer than the sum of the Pauling single bond covalent radii (2.07[108]). This distance is however shorter than that reported previously for the same interaction in a couple of other organotellurium(IV) trihalides [$C_{12}H_9N_2TeCl_3$ (2.417 Å) [103] and $(C_9H_8NO_2)TeCl_3$ (2.402 Å)[140]], indicating a stronger interaction

between tellurium and nitrogen in this case. Interestingly, the 2-(2-pyridyl)phenyl ligand appears to be held in an essentially planar geometry by this interaction between tellurium and the nitrogen in the pyridyl ring (Table 3.9). The slight deviation from planarity of the organic ligand is illustrated by the angle of 6.2(3)° between the mean planes of the pyridyl and phenyl rings. Indeed the whole molecule is planar apart from the apical bromine atoms, Br(1) and Br(2) to within ± 0.15 Å.

The tellurium bromine distances (2.589(3) Å equatorial, 2.673 (3) and 2.658(3) Å axial) fall within a range commonly found for such bonds[133,135,139,141,142] and are in good agreement with the sum of the single bond covalent radii [$Te_{(equatorial)}$- Br 2.51 Å and $Te_{(axial)}$- Br 2.67 Å][40.108]. There is a weak secondary intermolecular interaction between centrosymmetrically related molecules, Te...Br 3.596(3) Å. This type of interaction is a feature which has been noted previously in other organotellurium(IV) bromides, $C_{12}H_9TeBr_3$ (Te...Br, 3.71 Å)[135] and $C_4H_8STeBr_2$ (Te...Br, 3.59 Å)[141]. 2-(2-pyridyl)phenyltellurium(IV) tribromide can therefore be considered as essentially monomeric with a weak secondary interaction between tellurium and a neighbouring molecule bromine.

Table 3.7. Fractional atomic coordinates ($x10^4$ for all non-hydrogen atoms) with e.s.d.'s in parentheses for 2-(2-pyridyl)phenyltellurium(IV) tribromide.

| | x | y | z |
|---|---|---|---|
| Te | -1148( 2) | 4702( 1) | 6901( 1) |
| Br(1) | 373( 3) | 3242( 3) | 8772( 2) |
| Br(2) | -2577( 4) | 6522( 3) | 5122( 2) |
| Br(3) | -3547( 4) | 2395( 3) | 7016( 3) |
| N(1) | 504(22) | 6926(16) | 6923(14) |
| C(1) | -3102(29) | 5931(23) | 7944(17) |
| C(2) | -4963(30) | 5475(22) | 8358(19) |
| C(3) | -5976(34) | 6289(29) | 9061(19) |
| C(4) | -5318(34) | 7810(27) | 9306(19) |
| C(5) | -3481(28) | 8252(22) | 8884(16) |
| C(6) | -2333(29) | 7402(20) | 8135(17) |
| C(7) | -467(27) | 7924(21) | 7633(16) |
| C(8) | 423(33) | 9423(21) | 7677(23) |
| C(9) | 2327(35) | 9709(22) | 7082(25) |
| C(10) | 3045(40) | 8803(25) | 6354(24) |
| C(11) | 2176(31) | 7331(28) | 6366(18) |

Table 3.8. Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses for 2-(2-pyridyl)phenyltellurium(IV) tribromide .

| | | | | | |
|---|---|---|---|---|---|
| Te | ---Br(1) | 2.673 ( 3) | C(5) | ---C(6) | 1.410 (25) |
| Te | ---Br(2) | 2.658 ( 3) | C(6) | ---C(1) | 1.438 (25) |
| Te | ---Br(3) | 2.589 ( 3) | C(6) | ---C(7) | 1.415 (28) |
| Te | ---C(1) | 2.110 (19) | C(8) | ---C(9) | 1.435 (34) |
| Te | ---N(1) | 2.244 (14) | C(8) | ---C(7) | 1.441 (25) |
| C(2) | ---C(3) | 1.312 (29) | C(9) | ---C(10) | 1.312 (35) |
| C(2) | ---C(1) | 1.373 (29) | C(10) | ---C(11) | 1.397 (28) |
| C(3) | ---C(4) | 1.471 (29) | C(11) | ---N(1) | 1.295 (25) |
| C(4) | ---C(5) | 1.357 (30) | N(1) | ---C(7) | 1.407 (21) |

| | | | | | |
|---|---|---|---|---|---|
| Br(1) | -Te -Br(2) | 172.4 ( 1) | C(5) | -C(6) -C(7) | 121.7 (16) |
| Br(1) | -Te -Br(3) | 93.5 ( 1) | C(1) | -C(6) -C(7) | 121.2 (17) |
| Br(2) | -Te -Br(3) | 92.8 ( 1) | Te | -C(1) -C(2) | 125.4 (16) |
| Br(1) | -Te -C(1) | 87.5 ( 5) | Te | -C(1) -C(6) | 112.8 (14) |
| Br(2) | -Te -C(1) | 88.1 ( 5) | C(2) | -C(1) -C(6) | 121.7 (18) |
| Br(3) | -Te -C(1) | 92.5 ( 6) | C(9) | -C(8) -C(7) | 117.4 (20) |
| Br(1) | -Te -N(1) | 88.4 ( 4) | C(8) | -C(9) -C(10) | 120.2 (21) |
| Br(2) | -Te -N(1) | 84.7 ( 4) | C(9) | -C(10) -C(11) | 119.2 (23) |
| Br(3) | -Te -N(1) | 170.4 ( 4) | C(10) | -C(11) -N(1) | 123.4 (22) |
| C(1) | -Te -N(1) | 78.2 ( 7) | Te | -N(1) -C(11) | 126.8 (14) |
| C(3) | -C(2) -C(1) | 118.9 (21) | Te | -N(1) -C(7) | 113.0 (11) |
| C(2) | -C(3) -C(4) | 122.9 (21) | C(11) | -N(1) -C(7) | 120.3 (17) |
| C(3) | -C(4) -C(5) | 117.0 (20) | C(6) | -C(7) -C(8) | 127.1 (17) |
| C(4) | -C(5) -C(6) | 121.5 (19) | C(6) | -C(7) -N(1) | 114.7 (15) |
| C(5) | -C(6) -C(1) | 117.0 (17) | C(8) | -C(7) -N(1) | 117.8 (18) |

TABLE 3.9

**Mean Plane Analysis for**

**2-(2-Pyridyl)phenyltellurium(IV) tribromide**

Phenyl Ring

| Perp. Dist. to Plane | |
|---|---|
| 0.02940 | C1 |
| -0.03283 | C2 |
| 0.03034 | C3 |
| -0.02615 | C4 |
| 0.02496 | C5 |
| -0.02572 | C6 |

Mean Dev=0.028234.

RMS Dev=0.028376.

Pyridyl Ring

| Perp. Dist. to Plane | |
|---|---|
| 0.00471 | N1 |
| 0.00154 | C7 |
| -0.03668 | C8 |
| 0.06948 | C9 |
| -0.06390 | C10 |
| 0.02486 | C11 |

Mean Dev=0.033525

RMS Dev=0.042617

Equatorial Plane through Molecule

| Perp. Dist. to Plane | |
|---|---|
| 0.13235 | Te |
| 0.08726 | Br3 |
| 0.04589 | C1 |
| -0.08677 | C2 |
| -0.03545 | C3 |
| -0.03269 | C4 |
| 0.08779 | C5 |
| 0.05229 | C6 |
| 0.08685 | C7 |
| -0.00743 | C8 |
| 0.02473 | C9 |
| -0.12412 | C10 |
| 0.02029 | C11 |
| 0.06847 | N1 |

Mean Dev=0.099446 RMS Dev=0.128813

The remainder of the atoms, Br(1) and Br(2) lie at the following distances perpendicular to the plane; Br(1) 2.79330 and Br(2) -2.51938

Torsion Angles.

| | |
|---|---|
| N1-C7-C6-C5 | 178.9 |
| C8-C7-C6-C1 | 174.8 |
| N1-C7-C6-C1 | 2.0 |
| C8-C7-C6-C5 | -8.3 |

Figure 3.5. The structure of 2-(2-pyridyl)phenyltellurium(IV)tribromide showing the atom numbering.

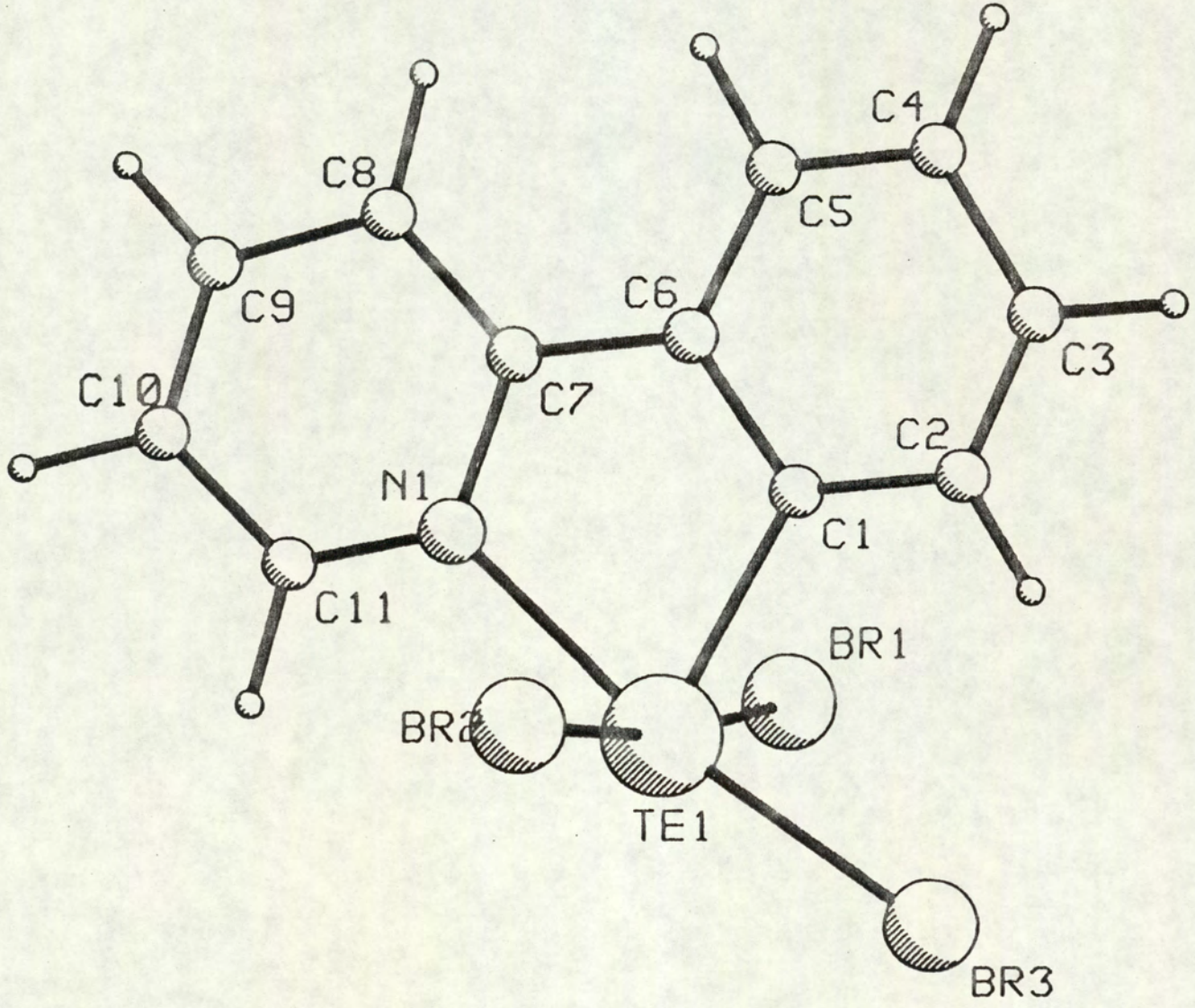
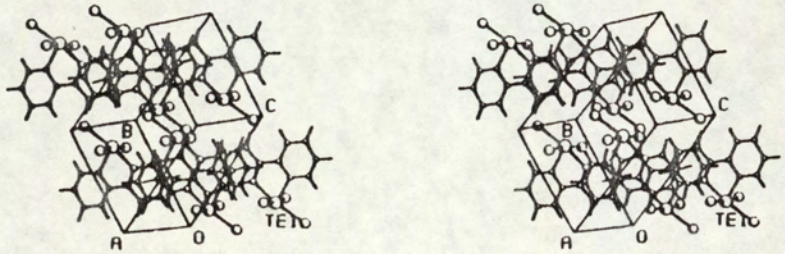


Figure 3.6. Stereoscopic packing diagram of 2-(2-pyridyl)phenyltellurium(IV) tribromide.



### 3.2.2 (2-N,N-Dimethylbenzylamine-C,N') Tellurium (IV) tribromide, (4)

The structural elucidation of this material was undertaken to examine the secondary bonding[39], if any, of an aliphatic nitrogen with tellurium in an $RTeX_3$ system and so provide a direct comparison with the structural study of 2-(2-pyridyl) phenyltellurium(IV) tribromide, in which the donating nitrogen atom was in an aromatic environment.

The material was supplied in a powdered form by Dr H B Singh, of the Indian Institute of Technology, Bombay. Dark brown crystals were grown from a saturated medium of nitromethane. The crystal chosen for X-ray analysis was 0.28 x 0.38 x 0.60 mm.

Elemental Analysis: Theoretical percentage C 21.4 N 2.78 H 2.41, Actual percentage C 21.3 N 2.80 H 2.39.

**Crystal Data**

Molecular formula $C_9H_{12}NTeBr_3$, Mr = 501.5, monoclinic, space group $P2_1/n$, a = 9.226(6) Å, b = 15.439(8) Å, c = 9.618(5) Å, ß = 94.94°(5), U = 1364.9 $Å^3$, Z = 4, $D_C$ = 2.441 $gcm^{-3}$, F(000) = 920, $\lambda$(Mo-$K_\alpha$) = 0.71069 Å, $\mu$ = 11.530 $mm^{-1}$.

2412 unique reflections were scanned in the data collection range of 2-25θ. From this original data set, 938 significant reflections with $I>2.5\sigma(I)$ were used as the bases for the structural analysis. Termination of the computer analysis stage occurred when all shifts were less than 0.01σ and R and Rw were

4.19% and 5.61% respectively, with $w=1/[\sigma 2(F) + 0.011F^2]$

Final atomic coordinates for (2-N,N-dimethylbenzylamine-C,N')tellurium(IV) tribromide are given in table 3.10, with bond distances, bond angles and mean plane calculations presented in tables 3.11 and 3.12 respectively.

If the intramolecular Te...N interaction is considered to be significant, the environment around tellurium can be considered to a first approximation as pseudo octahedral. In this representation the axial positions are occupied by Br(1) and Br(2) while C(1), N(1), Br(3) and the tellurium lone pair of electrons occupy the equatorial positions. The distortion from ideal octahedral geometry, particularly N(1)-Te-Br(3), [167.4(3)°] can be explained in terms of a lone pair of electrons occupying an equatorial position between N(1) and Br(3), whilst the small value of the angle N(1)-Te-C(1) [76.1(5)°] is due to the constraints imposed on the system by the five membered endocyclic ring. These values are analogous to those found in the crystal structures of (2-phenylazophenyl-C,N')tellurium(IV) trichloride[103] and 2-(2-pyridyl)phenyltellurium(IV) tribromide (this study). The molecular and unit cell packing diagrams are shown in figures 3.7 and 3.8 respectively.

The apical Te-Br bond lengths [2.758(2), 2.633(3) Å] span the value for single bond Pauling covalent radii, [Axial Te-Br 2.67 Å [40,108]]. The Te-Br distance at 2.633(3) Å falls within the range commonly observed for this bond,[135,139,143] however, the longer axial Te-Br distance is significantly displaced from these

more usual values. The Te-Br equatorial bond at 2.632(3) Å is also elongated relative to Pauling's theoretical value (2.51 Å)[40,108] and values commonly found for this bond [135,139,142]. The reason for the relatively long equatorial Te-Br bond and the disparity between the two axial Te-Br lengths is unclear, never-the-less some information may be provided by the studies of 2-biphenyltellurium triiodide[137] and phenyltellurium trichloride[87]. In both structures there is a pronounced lengthening of one of the tellurium halide bonds, leading the authors in each case to conclude that the structure had a tendency to exist as a telluronium salt of the type $[RX_2Te^+]X^-$. Indeed this tendency goes much further in solution, as shown by the conductivity of organotellurium trihalides in polar non-aqueous solutions[144].

In this structure there is a very weak secondary intermolecular contact of 3.869 Å between Te...Br(1)(2-x,1-y,1-z). This distance although only slightly less than the sum of van der Waals radii [3.91 Å][107] may still be a contributing factor in causing the long Te-Br(1) bond distance. There are no other significant intermolecular distances shorter than the sum of the relevant van der Waals radii, and so (2-N,N-dimethylbenzylamine-C,N')tellurium(IV) tribromide is therefore essentially monomeric. The distance Te-C(1) [2.121(14) Å] is in good agreement with typical values found in comparable $Te^{IV}$ complexes; eg (bip)$TeBr_3$[135] (bip=2-biphenylyl), $C_4H_8STeBr_2$[141], and (pap) $TeCl_3$ (pap= 2-phenylazophenyl)[103], where Te-C range in value from 2.11 - 2.16 Å and with the sum of

the Pauling single bond covalent radii[108]. The phenyl ring bonds and angles are unexceptional and are detailed in table 3.11. The phenyl ring itself is planar to within ±0.02 Å.

The Te-N interaction at 2.422(14) Å is significantly longer than the sum of the Pauling single bond covalent radii[108]. This value is never-the-less akin to the values reported for secondary Te...N interactions in (pap)Te-$Cl_3$, 2.417 Å[103] and (dapy) $TeCl_3$ (dapy=2,6-diacetylIpyridine), 2.402 Å[140]. An additional point of interest is that this distance is longer than the $Te\text{-}N_{(aromatic)}$ distance found in 2-(2-pyridyl)phenyltellurium(IV) tribromide [2.244(14) Å], indicating a weaker interaction in this case.

Table 3.10, Fractional atomic coordinates ($x10^4$ for all non-hydrogen atoms) with e.s.d.'s in parentheses for (2-N,N-dimethylbenzylamine-C,N')tellurium(IV) tribromide .

| | x | y | z |
|---|---|---|---|
| Te(1) | 1383( 1) | 3822( 1) | 4315( 1) |
| Br(1) | -1558( 2) | 3743( 1) | 4624( 2) |
| Br(2) | 4195( 3) | 3929( 1) | 4050( 3) |
| Br(3) | 799( 3) | 3836( 1) | 1587( 2) |
| N(1) | 1713(15) | 3489( 9) | 6782(14) |
| C(1) | 1523(16) | 2451( 9) | 4374(17) |
| C(2) | 1788(19) | 1944(10) | 3231(18) |
| C(3) | 1843(21) | 1062(10) | 3352(22) |
| C(4) | 1707(23) | 676(11) | 4615(22) |
| C(5) | 1415(22) | 1168(11) | 5811(23) |
| C(6) | 1310(19) | 2077( 9) | 5675(20) |
| C(7) | 1008(20) | 2635(10) | 6891(18) |
| C(8) | 3300(21) | 3389(12) | 7295(24) |
| C(9) | 1069(28) | 4177(12) | 7569(20) |

Table 3.11. Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses for (2-N,N-dimethylbenzylamine-C,N')tellurium(IV)tribromide .

| | | | | | |
|---|---|---|---|---|---|
| Te(1) ---Br(1) | 2.758 ( 2) | C(1) ---C(2) | 1.388 (21) | | |
| Te(1) ---Br(2) | 2.633 ( 3) | C(1) ---C(6) | 1.407 (21) | | |
| Te(1) ---Br(3) | 2.632 ( 2) | C(2) ---C(3) | 1.367 (20) | | |
| Te(1) ---N(1) | 2.422 (14) | C(3) ---C(4) | 1.368 (24) | | |
| Te(1) ---C(1) | 2.121 (14) | C(4) ---C(5) | 1.423 (25) | | |
| N(1) ---C(7) | 1.478 (20) | C(5) ---C(6) | 1.412 (22) | | |
| N(1) ---C(8) | 1.512 (22) | C(6) ---C(7) | 1.498 (22) | | |
| N(1) ---C(9) | 1.460 (22) | | | | |
| Br(1) -Te(1) -Br(2) | 178.7 ( 1) | C(7) -N(1) -C(9) | 114.3 (14) | | |
| Br(1) -Te(1) -Br(3) | 89.4 ( 1) | C(8) -N(1) -C(9) | 109.3 (15) | | |
| Br(2) -Te(1) -Br(3) | 91.3 ( 1) | Te(1) -C(1) -C(2) | 123.8 (12) | | |
| Br(1) -Te(1) -N(1) | 85.8 ( 3) | Te(1) -C(1) -C(6) | 114.9 (11) | | |
| Br(2) -Te(1) -N(1) | 93.8 ( 3) | C(2) -C(1) -C(6) | 121.3 (14) | | |
| Br(3) -Te(1) -N(1) | 167.4 ( 3) | C(1) -C(2) -C(3) | 120.2 (17) | | |
| Br(1) -Te(1) -C(1) | 90.6 ( 4) | C(2) -C(3) -C(4) | 120.2 (18) | | |
| Br(2) -Te(1) -C(1) | 90.4 ( 4) | C(3) -C(4) -C(5) | 121.5 (16) | | |
| Br(3) -Te(1) -C(1) | 92.4 ( 4) | C(4) -C(5) -C(6) | 118.2 (18) | | |
| N(1) -Te(1) -C(1) | 76.1 ( 5) | C(1) -C(6) -C(5) | 118.4 (17) | | |
| Te(1) -N(1) -C(7) | 103.9 (10) | C(1) -C(6) -C(7) | 120.5 (14) | | |
| Te(1) -N(1) -C(8) | 112.2 (11) | C(5) -C(6) -C(7) | 121.0 (17) | | |
| C(7) -N(1) -C(8) | 107.8 (13) | N(1) -C(7) -C(6) | 110.2 (13) | | |
| Te(1) -N(1) -C(9) | 109.3 (10) | | | | |

TABLE 3.12

**Mean Plane Analysis for (2-N,N-dimethylbenzylamine-C,N')tellurium (IV) tribromide**

Phenyl Ring

| Perpendicular Distance to Plane | |
|---|---|
| 0.00973 | C1 |
| 0.00770 | C2 |
| -0.02107 | C3 |
| 0.01636 | C4 |
| 0.00113 | C5 |
| -0.00113 | C6 |

Mean Dev=0.011643. RMS Dev=0.013286

Equatorial Plane Through Molecule

| Perpendicular Distance to Plane | |
|---|---|
| 0.16406 | Te1 |
| -0.35776 | Br3 |
| 0.40695 | N1 |
| 0.08895 | C1 |
| 0.26067 | C2 |
| 0.18016 | C3 |
| -0.00647 | C4 |
| -0.20535 | C5 |
| -0.16633 | C6 |
| -0.36489 | C7 |

Mean Dev=0.220160. RMS Dev=0.251367

The remainder of the atoms within the molecule lie at the following distances perpendicular to this plane, Br1 -2.54091, Br2 2.75446, C8 1.84270, and C9 -0.08390.

Figure 3.7. The structure of (2-N,N-dimethylbenzylamine-C,N')tellurium(IV)tribromide showing the atom numbering.

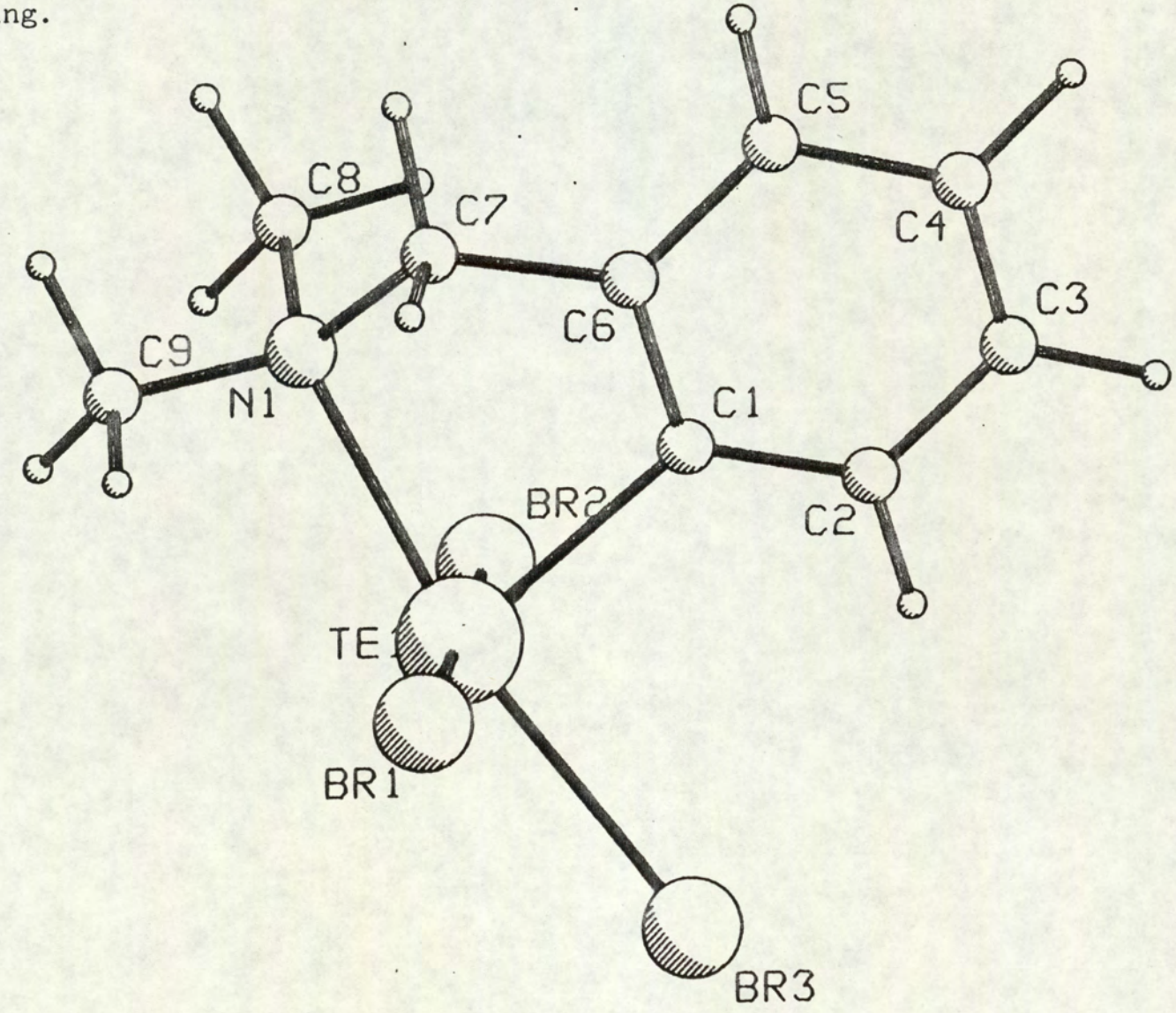
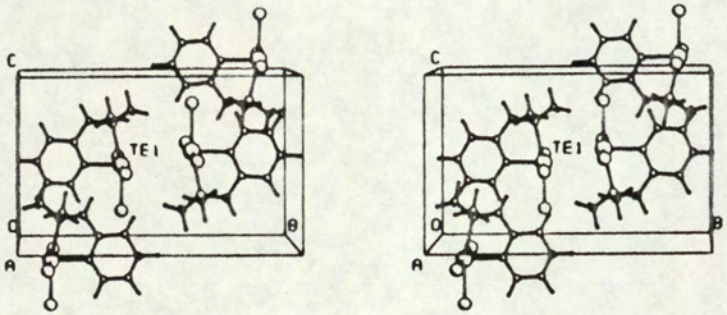


Figure 3.8. Stereoscopic packing diagram of (2-N,N-dimethylbenzylamine-C,N')tellurium(IV)tribromide





### 3.2.3 2-Dichloro(butyl)tellurobenzaldehyde, (5)

Unlike the other structures studied, 2-dichloro(butyl)-tellurobenzaldehyde contains a potentially electron donating oxygen atom rather than a nitrogen atom in one of its organic ligands. The examination of this structure, never-the-less afforded an opportunity to explore the preference of tellurium(IV) for forming inter- as opposed to intra-molecular secondary bonds in an $R_2TeX_2$ system. In addition, the study of 2-dichloro(butyl)tellurobenzaldehyde provided a chance to assess the effect of the environment around tellurium, on the anticipated tellurium-oxygen interaction.

Colourless needle-like crystals were supplied for analysis by K Y Abid of this department.

**Crystal Data**

$C_{11}H_{14}OCl_2Te$, Mr = 360.7, crystal size 0.58 x 0.28 x 0.03 mm (second crystal), monoclinic $P2_1/n$, a = 21.760(9) Å, b = 8.178(8) Å, c =15.441(5) Å. ß = 92.76(3)°, U = 2744.6Å$^3$, Z = 8, $D_c$ = 1.746 gcm$^{-3}$, F(000) = 1392, $\lambda$(Mo-k$_\alpha$) = 0.71069Å, $\mu$ = 2.61 mm$^{-1}$.

Total data measured 5587 reflections; significant data [I>2.5θ(I)] 3099 reflections. Least squares refinement was terminated when all shifts were less than 0.01σ and R and $R_w$ were 6.26% and 8.44% respectively; $w=1/(\sigma^2(F) + 0.002F^2)$.

2-Dichloro(butyl)tellurobenzaldehyde appears to suffer severe decomposition when irradiated with X-rays, this necessitated the replacement of the original crystal during analysis, once the intensity standards had fallen to sixty per cent

of their initial value. Before Lorentz and polarisation factors were applied, scale factors were invoked to correct for crystal deterioration and to equalize the data collected from the two crystals used. Similar sensitivity of diorganyltellurium(IV) dichlorides to X-rays has been noted previously, during the studies of di-*cis*-2-chlorocyclohexyldichlorotellurium(IV) and *p*-tolyl-2-chlorocyclohexyldichlorotellurium(IV) [145,146].

Final atomic coordinates for 2-dichloro(butyl)telluro-benzaldehyde are given in table 3.13, with bond distances, bond angles and mean plane calculations given in tables 3.14 and 3.15 respectively. A view of the molecule and a stereoscopic packing diagram are shown in figures 3.9 and 3.10.

Within the asymmetric unit there are two discrete molecules which are approximately related by a non-crystallographic pseudo two fold screw axis. The geometries of the two distinct molecules are quite similar, therefore only molecule B is shown in figure 3.9. If the secondary interaction between tellurium and oxygen is considered to be significant, the basic coordination around the central tellurium atom in each molecule can best be described as essentially octahedral. In this representation of the coordination sphere around tellurium, the axial sites are occupied by the two chlorine atoms which are covalently bonded to the tellurium, and the equatorial plane consists of two carbon atoms covalently bound to tellurium, an oxygen atom which is linked to tellurium via secondary bonding[39] and the lone pair of electrons from tellurium. Some of the distortions of the coordination geometry from idea octahedral

values (180° and 90°) can be explained in terms of valence shell electron pair repulsion theory, (VSEPR)[77,78] whilst others are due to constraints arising from the five membered chelate ring. The Cl-Te-C angles are marginally less than 90°, which suggests that the Te-Cl bonds are directed slightly away from tellurium's lone pair of electrons, as might be anticipated from VSEPR theory[77,78], giving a Cl-Te-Cl angle of 172.4(1)° in both molecules.

Each molecule can be considered as practically planar (deviation from planarity ±0.19 Å in molecule A and ±0.21 Å in molecule B) apart from the apical chlorine atoms which are situated above and below the plane of the molecule. If consideration is given to the atoms which form the basal plane of the molecule, Te, C(1), O(1), C(8), it is apparent that this group of atoms is coplanar to within ±0.05 Å in molecule A and ±0.08 Å in molecule B. In each instance the apical chlorines Cl(1) and Cl(2) lie on either side of this plane at a distance of 2.45 and 2.54 Å in molecule A and 2.45 and 2.56 Å in molecule B respectively; (table 3.15). The angle between the mean planes of the aromatic ligand and the butyl chain is 8.3° in molecule A and 11.0° in molecule B.

The distance Te-C(1) [2.121(11), 2.125(11) Å] is in good agreement with the sum of the Pauling single bond covalent radii for tellurium (1.37 Å) and an $sp^2$ hybridised carbon atom (0.74 Å)[108], and with values in the crystal structures of a number of analogous compounds, diphenyltellurium dichloride [87], dichloro-[(4)4-dimethyl-5-oxo-2,3,4,5-tetrahydro-2-furyl)methyl]-(4-methoxyphenyl)tellurium(IV)[147], bis(*p*-bromophenyl)

tellurium(IV) dichloride[85], range 2.094-2.136 Å. The Te-C(8) distance [2.124(12), 2.119(12) Å] is slightly shorter than the sum of the Pauling single bond covalent radii for tellurium and a $sp^3$ hybridised carbon atom (2.142 Å)[108], never-the-less, this distance is comparable with values reported in a number of $R_2TeX_2$ structures which fall in the range 2.11-2.16 Å[148-150].

The Te-O(1) distance (2.848(4), 2.830(4) Å) is much longer than the sum of the covalent radii (2.03 Å) [108], but is significantly shorter than the sum of the van der Waals radii (3.60 Å)[107]. This indicates that potentially, there is a significant secondary interaction existing between tellurium and oxygen. Similar long secondary Te...O contacts have been reported in a variety of tellurium(IV) compounds. For example, 2,6-diacetylpyridine(C,N,O)tellurium(IV) trichloride (Te...O 2.878 Å)[140], 10-acetonyl- phenoxatellurine Nitrate. (Te...O 2.775(3) Å) [151] triphenyl-telluronium cyanate-chloroform($^1/_2$). (Te...O 2.891(9), 3.005(9) Å)[152] and diphenyltellurium ditrifluoacetate (Te...O intermolecular 2.99-3.08 Å and intramolecular 3.0 - 3.14) (153).

The Te-Cl distances (2.503(4) - 2.512 (4) Å) are in good agreement with the sum of the single bond covalent radii ($Te_{(axial)}$[40]-Cl[108], 2.52 Å). These distances also fall within a range for $Te_{(axial)}$-Cl single covalent bonds found in the crystal structures of a number of other $R_2TeCl_2$ compounds (85-87,150,154). There is within the asymmetric unit, a weak secondary intermolecular interaction[39] between Te and Cl(1),

3.740(4), 3.775(4), which lies approximately *trans* to the Te-C(1). This feature of secondary Te...Cl intermolecular interactions occurs in a number of similar complexes (86,87,149). 2-Dichloro(butyl)tellurobenzaldehyde can therefore be considered as monomeric with a very weak secondary interaction between tellurium and a neighbouring chlorine. There are no other significant inter molecular distances within the unit cell, less than the sum of the van der Waals radii.

Table 3.13. Fractional atomic coordinates (x10$^5$ for tellurium and x10$^4$ all other non-hydrogen atoms ) with e.s.d.'s in parentheses for 2-dichloro(butyl)tellurobenzaldehyde

| | x | y | z |
|---|---|---|---|
| Te(1A) | 16955( 3) | 20227( 9) | 4766( 6) |
| Cl(1A) | 2408( 2) | 2386( 4) | -734( 3) |
| Cl(2A) | 989( 2) | 2064( 6) | 1708( 3) |
| O(1A) | 789( 5) | 794(13) | -747( 9) |
| C(1A) | 1161( 5) | 3946(13) | -92( 8) |
| C(2A) | 1281( 6) | 5536(17) | 173( 9) |
| C(3A) | 933( 6) | 6814(16) | -180( 9) |
| C(4A) | 465( 6) | 6533(19) | -792( 9) |
| C(5A) | 355( 6) | 4955(17) | -1060( 9) |
| C(6A) | 699( 6) | 3660(16) | -707( 8) |
| C(7A) | 553( 7) | 2024(20) | -1009(11) |
| C(8A) | 2324( 5) | 3461(15) | 1247( 9) |
| C(9A) | 2827( 6) | 2415(18) | 1679(10) |
| C(10A) | 3254( 6) | 3451(17) | 2250( 8) |
| C(11A) | 3796( 7) | 2422(21) | 2626(10) |
| Te(1B) | 83608( 3) | 64597( 9) | 45051( 6) |
| Cl(1B) | 7639( 2) | 6789( 4) | 5708( 3) |
| Cl(2B) | 9092( 2) | 6538( 5) | 3295( 3) |
| O(1B) | 9236( 5) | 5149(12) | 5730( 8) |
| C(1B) | 8897( 5) | 8350(14) | 5108( 8) |
| C(2B) | 8785( 6) | 9972(13) | 4873( 8) |
| C(3B) | 9130( 6) | 11169(15) | 5258( 9) |
| C(4B) | 9573( 6) | 10863(16) | 5881( 8) |
| C(5B) | 9674( 6) | 9269(18) | 6151( 9) |
| C(6B) | 9338( 5) | 7998(14) | 5756( 9) |
| C(7B) | 9477( 8) | 6340(17) | 6040(11) |
| C(8B) | 7743( 5) | 7933(15) | 3744( 9) |
| C(9B) | 7222( 5) | 6954(14) | 3323( 9) |
| C(10B) | 6786( 5) | 8004(15) | 2794( 8) |
| C(11B) | 6247( 6) | 7044(18) | 2379( 9) |

Table 3.14. Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses for 2-dichloro(butyl)tellurobenzaldehyde.

| | | | |
|---|---|---|---|
| Te(1A)---Cl(1A) | 2.504 ( 4) | Te(1B)---Cl(1B) | 2.504 ( 4) |
| Te(1A)---Cl(2A) | 2.503 ( 4) | Te(1B)---Cl(2B) | 2.512 ( 4) |
| Te(1A)...O(1A) | 2.848 ( 4) | Te(1B)...O(1B) | 2.830 ( 4) |
| Te(1A)---C(1A) | 2.121 (11) | Te(1B)---C(1B) | 2.125 (11) |
| Te(1A)---C(8A) | 2.124 (12) | Te(1B)---C(8B) | 2.119 (12) |
| O(1A) ---C(7A) | 1.192 (20) | O(1B) ---C(7B) | 1.196 (18) |
| C(1A) ---C(2A) | 1.384 (17) | C(1B) ---C(2B) | 1.393 (15) |
| C(1A) ---C(6A) | 1.369 (16) | C(1B) ---C(6B) | 1.383 (16) |
| C(2A) ---C(3A) | 1.387 (19) | C(2B) ---C(3B) | 1.353 (17) |
| C(3A) ---C(4A) | 1.374 (19) | C(3B) ---C(4B) | 1.352 (18) |
| C(4A) ---C(5A) | 1.373 (21) | C(4B) ---C(5B) | 1.384 (20) |
| C(5A) ---C(6A) | 1.393 (18) | C(5B) ---C(6B) | 1.395 (18) |
| C(6A) ---C(7A) | 1.447 (21) | C(6B) ---C(7B) | 1.452 (18) |
| C(8A) ---C(9A) | 1.519 (18) | C(8B) ---C(9B) | 1.509 (17) |
| C(9A) ---C(10A) | 1.509 (18) | C(9B) ---C(10B) | 1.494 (17) |
| C(10A)---C(11A) | 1.541 (20) | C(10B)---C(11B) | 1.526 (18) |

| | | | |
|---|---|---|---|
| Cl(1A)-Te(1A)-Cl(2A) | 172.4 ( 1) | Cl(1B)-Te(1B)-Cl(2B) | 172.4 ( 1) |
| Cl(1A)-Te(1A)-C(1A) | 87.1 ( 3) | Cl(1B)-Te(1B)-C(1B) | 87.0 ( 3) |
| Cl(2A)-Te(1A)-C(1A) | 87.7 ( 3) | Cl(2B)-Te(1B)-C(1B) | 87.3 ( 3) |
| Cl(1A)-Te(1A)-C(8A) | 87.0 ( 4) | Cl(1B)-Te(1B)-C(8B) | 86.9 ( 4) |
| Cl(2A)-Te(1A)-C(8A) | 88.2 ( 4) | Cl(2B)-Te(1B)-C(8B) | 88.9 ( 4) |
| O(1A) -Te(1A)-C(1A) | 68.8 ( 4) | O(1B) -Te(1B)-C(1B) | 69.2 ( 4) |
| O(1A) -Te(1A)-C(8A) | 166.8 ( 4) | O(1B) -Te(1B)-C(8B) | 167.2 ( 4) |
| C(1A) -Te(1A)-C(8A) | 98.4 ( 4) | C(1B) -Te(1B)-C(8B) | 98.6 ( 4) |
| Te(1A)-C(1A) -C(2A) | 118.8 ( 8) | Te(1B)-C(1B) -C(2B) | 119.6 ( 8) |
| Te(1A)-C(1A) -C(6A) | 122.0 ( 8) | Te(1B)-C(1B) -C(6B) | 120.8 ( 8) |
| C(2A) -C(1A) -C(6A) | 119.2 (11) | C(2B) -C(1B) -C(6B) | 119.5 (10) |
| C(1A) -C(2A) -C(3A) | 120.0 (12) | C(1B) -C(2B) -C(3B) | 119.2 (11) |
| C(2A) -C(3A) -C(4A) | 121.1 (13) | C(2B) -C(3B) -C(4B) | 122.7 (12) |
| C(3A) -C(4A) -C(5A) | 118.6 (13) | C(3B) -C(4B) -C(5B) | 119.1 (12) |
| C(4A) -C(5A) -C(6A) | 120.9 (12) | C(4B) -C(5B) -C(6B) | 119.9 (11) |
| C(1A) -C(6A) -C(5A) | 120.3 (12) | C(1B) -C(6B) -C(5B) | 119.5 (11) |
| C(1A) -C(6A) -C(7A) | 121.6 (12) | C(1B) -C(6B) -C(7B) | 122.7 (11) |
| C(5A) -C(6A) -C(7A) | 118.1 (12) | C(5B) -C(6B) -C(7B) | 117.8 (12) |
| O(1A) -C(7A) -C(6A) | 125.9 (14) | O(1B) -C(7B) -C(6B) | 124.0 (14) |
| Te(1A)-C(8A) -C(9A) | 111.4 ( 9) | Te(1B)-C(8B) -C(9B) | 112.3 ( 8) |
| C(8A) -C(9A) -C(10A) | 110.5 (11) | C(8B) -C(9B) -C(10B) | 112.0 (10) |
| C(9A) -C(10A)-C(11A) | 110.6 (12) | C(9B) -C(10B)-C(11B) | 112.9 (10) |

**TABLE 3.15**

**Mean Plane Analysis for 2-Dichloro(butyl)tellurobenzaldehyde**

Equatorial Plane Through Whole Molecule

| Molecule A | | Molecule B | |
|---|---|---|---|
| Perpendicular Distance to Plane | | Perpendicular Distance to Plane | |
| 0.14843 | Te1 | -0.19708 | Te1 |
| 0.01306 | O1 | -0.01739 | O1 |
| 0.04402 | C1 | -0.08872 | C1 |
| 0.07355 | C2 | -0.10994 | C2 |
| 0.02401 | C3 | -0.04596 | C3 |
| -0.04925 | C4 | 0.07395 | C4 |
| -0.09714 | C5 | 0.15649 | C5 |
| -0.04101 | C6 | 0.06080 | C6 |
| -0.07526 | C7 | 0.10347 | C7 |
| 0.10045 | C8 | -0.16243 | C8 |
| 0.00974 | C9 | -0.00971 | C9 |
| 0.03184 | C10 | 0.02574 | C10 |
| -0.18243 | C11 | 0.21079 | C11 |
| Mean Dev=0.068475. | | Mean Dev=0.097113 | |
| RMS Dev=0.084949. | | RMS Dev=0.116733 | |

Basal Plane of the Molecule.

| Molelcule A | | Molecule B | |
|---|---|---|---|
| Perpendicular Distance to Plane | | Perpendicular Distance to Plane` | |
| 0.04950 | Te1 | -0.05792 | Te1 |
| -0.02517 | O1 | 0.02922 | O1 |
| 0.00750 | C1 | -0.00825 | C1 |
| -0.03183 | C8 | -0.03694 | C8 |
| Mean Dev=0.028502. | | Mean Dev=0.03308 | |
| RMS Dev=0.032225 | | RMS Dev=0.037555 | |

Cl(1) and Cl(2) lie at the following distances perpendicular to this plane.

Cl(1) -2.45268 Cl(2) 2.54041 Cl(1) 2.44457 Cl(2)-2.55699

Torsion Angles (°)

| Molecule A | | Molecule B | |
|---|---|---|---|
| C(1)-C(6)-C(7)-0(1) | 2.9 | C(1)-C(6)-C(7)-0(1) | 1.8 |
| C(5)-C(6)-C(7)-0(1) | -177.1 | C(5)-C(6)-C(7)-0(1) | -177.2 |

Figure 3.9. The structure of 2-dichloro(butyl)tellurobenzaldehyde showing the atom numbering.

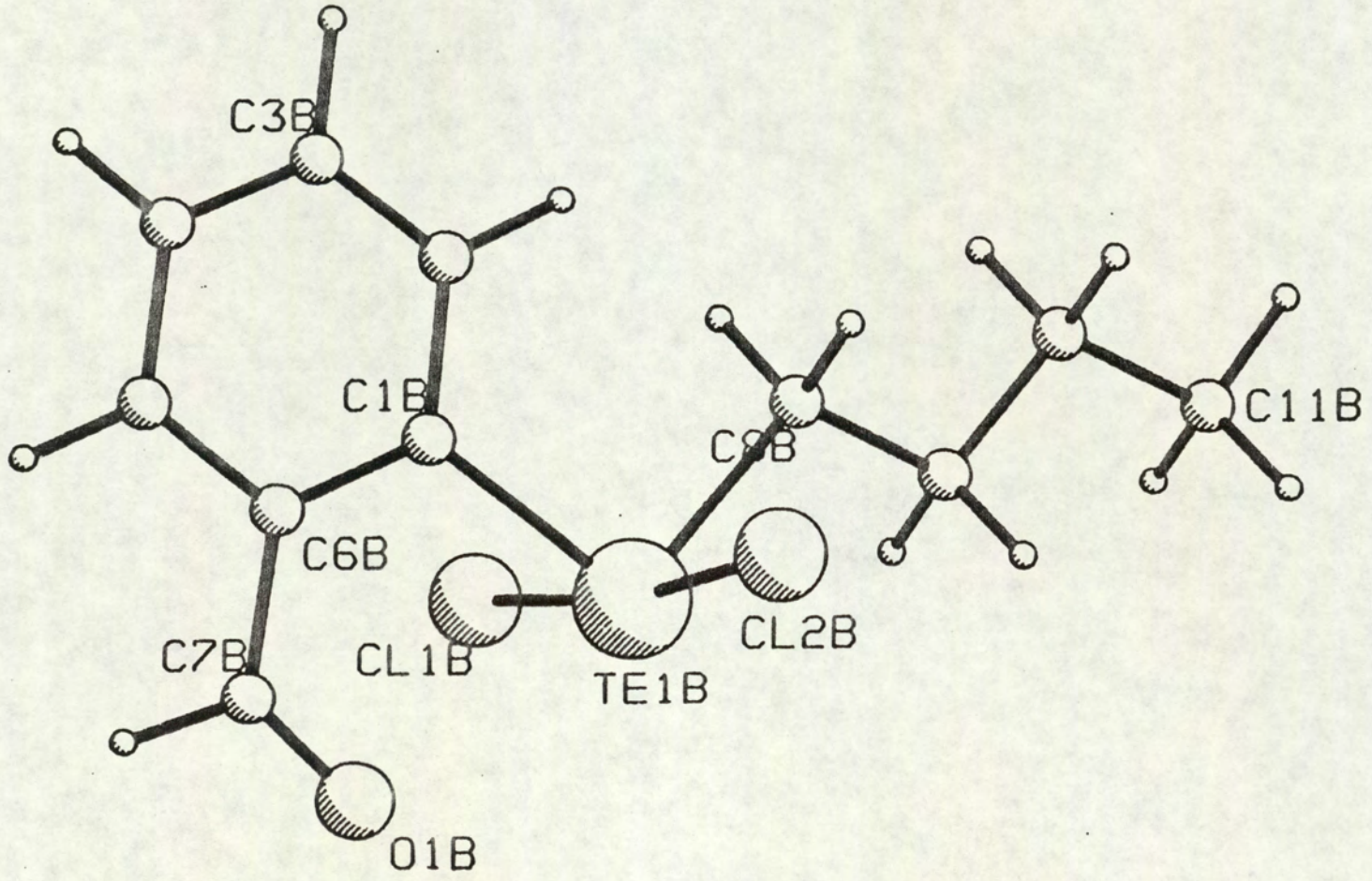
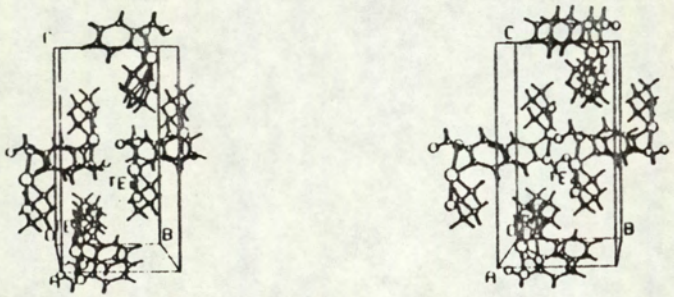


Figure 3.10. Stereoscopic packing diagram of 2-dichloro(butyl)tellurobenzaldehyde.



### 3.2.4 2 Dichloro(butyl)telluro-N-dimethylbenzylammonium Chloride, (6)

A sample of colourless crystals were supplied for examination by Dr H B Singh of the Indian Insitute of Technology in Bombay. It was believed at that time, that these were crystals of a diorganyltellurium(IV) dichloride with one of the organic ligands possessing an electron donating nitrogen. Based on this premise, the particular aim of this analysis was to examine the tellurium-nitrogen interaction in an $R_2TeX_2$ system. To observe how this interaction was effected, by the changing Lewis acidity of tellurium in this environment compared to that in an $RTeX_3$ system. It was additionally hoped that this supposed diorganyltellurium(IV) dichloride would provide an interesting comparison with 2-dichloro(butyl)tellurobenzaldehyde, analysed previously, where the donating atom was oxygen.

Single crystal X-ray analysis, revealed the colourless crystals to be a salt of the type $[R(R^+)TeCl_2]Cl^-$, rather than the anticipated and desired diorganyltellurium(IV) dichloride. This result was in turn confirmed by the elemental analysis (Found % C 36.3, H 5.18 N 3.43 Cl 25.1. Theoretical % C 36.7 H 5.21, N 3.29, Cl 24.3). The crystals supplied were in fact 2-dichloro(butyl)telluro-N-dimethylbenzylammonium chloride, which although not of direct relevance to the main theme of this study, still provided useful information on secondary bonding and on the arrangement of atoms around tellurium in a tellurium(IV) complex.

**Crystal Data**

$[C_{13}H_{22}Cl_2NTe]^+Cl^-$, Mr = 426.3, crystal size 0.08 x 0.13 x 0.47 mm, monoclinic $P2_1/n$, a = 14.278(12) Å, b = 8.349(5) Å, c = 14.480(8) Å, ß = 93.59(5), U = 1722.7 $Å^3$, Z = 4, $D_c$ = 1.644 $gcm^{-3}$, F(000) = 840, $\lambda$(Mo-$K_\alpha$) = 0.71069 Å, $\mu$ = 2.23 $mm^{-1}$. Unique data measured 3025 reflections; significant data [$I>2.5\theta(I)$] 2245 reflections.

With the exception of the hydrogen attached to the positively charged nitrogen atom, hydrogens were placed in calculated positions (C-H 1.08 Å), and allowed to "ride" on their respective carbon atoms in subsequent least-squares calculations. Least squares refinement was terminated when all shifts were less than 0.01$\sigma$ and R and $R_w$ were 2.88% and 3.60% respectively; $w=1/(\sigma^2(F) + 0.00055F^2)$.

Final atomic coordinates for 2 dichloro(butyl)telluro-N-dimethyl benzylammonium chloride are given in table 3.16, with bond distances, bond angles, and mean plane calculations given in tables 3.17 and 3.18 respectively. A view of the molelcule and a stereoscopic packing diagram are shown in figures 3.11 and 3.12.

The primary geometry about tellurium may be considered to be trigonal bipyramidal, with two chlorines covalently bound to the tellurium in the axial positions and two carbon atoms covalently bound to tellurium and the lone pair of electrons on tellurium occupying the equatorial positions. This representation

does not include the secondary interaction between tellurium and the chloride ion [Te...Cl$^-$ 3.337(2) Å]. If this interaction is considered significant, then an alternative description of the geometry around tellurium is pseudo-octahedral. In this approximation C(1) lies approximately *trans* to the chloride ion [C(1)-Te...Cl(3)$^-$ 173.3(2)°], and C(10) lies opposite the sterically active tellurium lone pair of electrons in the equatorial plane, with the two covalently bound chlorines occupying the axial sites. The latter description seems to be the more consistent of the two, with the bond angles of the structure given in table 3.17.

The axial Te-Cl distances 2.487(1) and 2.527(1) Å are in good agreement with the sum of the covalent radii for an axial Te-Cl bond [2.52 Å][40,108], and may be compared with a number of axial Te-Cl bonds, in analogous structures which fall in the range 2.45-2.57 Å (mean 2.51 Å)[155-157]. Within the asymmetric unit, there is a Te...Cl$^-$ contact of 3.337(2) Å. This distance lies between tellurium bridging chlorine distances found in triphenyltellurium chloride [3.142(1)-3.234(1) Å][158] and a secondary intermolecular contact indentified in dimethyl ammonium tetrachloro(*p*-phenoxyphenyl|)tellurate [3.619 Å][159], in addition this distance falls well within the sum of the van der Waals radii [3.81,[107] 4-00 Å[108]].

The Te-C(1), distance at 2.134(4)Å is slightly longer than the sum of the Pauling single bond covalent radii for Te-C $sp^2$ [2.11 Å][108], but is similar to values reported in other tellurium (IV) structural studies[85,155,157]. The Te-C(10) length at 2.154(4) Å is analogous to the theoretical value for the Pauling

single bond covalent radii for Te-$Csp^3$, and falls within a range for Te-$Csp^3$ bonds, defined by previously reported tellurium(IV) structures [Te-$Csp^3$ 2.128-2.177 Å](160). The phenyl ring of the N-dimethylbenzylammonium ligand is essentially planar (±0.006) (table 3.18). The mean plane of this moiety is orientated at an angle of 83.7(2)° to the mean plane of the butyl chain.

There is no secondary bonding between the protonated nitrogen and the central tellurium atom, as the protonated nitrogen is twisted away from the tellurium at a distance of 4.380(4) Å. The chloride ion, on the other hand, forms a hydrogen bond with the dimethyl benzylammonium group in the symmetry related molecule; [H...$Cl^-_{(0.5+X,\ 0.5-Y,\ 0.5+Z)}$ 2.327 Å]. This distance is akin, to that noted in diethylammonium tetrachloro (*p*-phenoxyphenyl)tellurate, where the shortest hydrogen bonding occurred between a diethylammonium group and a symmetry related chlorine. [H...$Cl_{(X,1+Y,Z)}$ 2.368 Å](159).

Table 3.16. Fractional atomic coordinates ($\times 10^5$ for tellurium and $\times 10^4$ for all other non-hydrogen atoms) with e.s.d.'s in parentheses for 2-Dichloro(butyl)telluro-N-dimethylbenzylammonium Chloride

| | x | y | z |
|---|---|---|---|
| Te(1) | 42016( 2) | 15017( 4) | 13165( 2) |
| Cl(1) | 4163( 1) | -1414( 2) | 848( 1) |
| Cl(2) | 3995( 1) | 4363( 2) | 1734( 1) |
| Cl(3) | 6343( 1) | 1511( 2) | 2381( 1) |
| N(1) | 3222( 2) | 3237( 5) | -1431( 2) |
| C(1) | 2773( 3) | 1567( 5) | 788( 3) |
| C(2) | 2064( 3) | 1469( 7) | 1408( 3) |
| C(3) | 1134( 3) | 1463( 7) | 1088( 4) |
| C(4) | 907( 3) | 1561( 7) | 150( 4) |
| C(5) | 1605( 3) | 1659( 6) | -462( 3) |
| C(6) | 2547( 3) | 1681( 5) | -158( 3) |
| C(7) | 3271( 3) | 1714( 6) | -875( 3) |
| C(8) | 3803( 4) | 3103( 8) | -2256( 3) |
| C(9) | 3506( 4) | 4658( 7) | -861( 4) |
| C(10) | 3964( 3) | 625( 7) | 2681( 3) |
| C(11) | 3769( 4) | 1819( 6) | 3427( 3) |
| C(12) | 3655( 4) | 968( 7) | 4334( 3) |
| C(13) | 3380( 4) | 2074( 9) | 5097( 4) |

Table 3.17. Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses for 2-Dichloro(butyl)telluro-N-dimethylbenzylammonium Chloride

| | | | |
|---|---|---|---|
| Te(1) ---Cl(1) | 2.527 ( 1) | C(2) ---C(3) | 1.379 ( 6) |
| Te(1) ---Cl(2) | 2.487 ( 1) | C(3) ---C(4) | 1.381 ( 8) |
| Te(1) ---C(1) | 2.134 ( 4) | C(4) ---C(5) | 1.377 ( 7) |
| Te(1) ---C(10) | 2.154 ( 4) | C(5) ---C(6) | 1.389 ( 6) |
| N(1) ---C(7) | 1.504 ( 6) | C(6) ---C(7) | 1.511 ( 6) |
| N(1) ---C(8) | 1.500 ( 6) | C(10) ---C(11) | 1.508 ( 7) |
| N(1) ---C(9) | 1.486 ( 6) | C(11) ---C(12) | 1.509 ( 7) |
| C(1) ---C(2) | 1.396 ( 6) | C(12) ---C(13) | 1.511 ( 8) |
| C(1) ---C(6) | 1.391 ( 6) | Te(1) ...Cl(3) | 3.338 ( 3) |
| | | | |
| Cl(1) -Te(1) -Cl(2) | 171.9 ( 1) | C(1) -C(2) -C(3) | 120.4 ( 4) |
| Cl(1) -Te(1) -C(1) | 85.6 ( 1) | C(2) -C(3) -C(4) | 119.5 ( 4) |
| Cl(2) -Te(1) -C(1) | 86.4 ( 1) | C(3) -C(4) -C(5) | 120.2 ( 4) |
| Cl(1) -Te(1) -C(10) | 85.2 ( 1) | C(4) -C(5) -C(6) | 121.5 ( 4) |
| Cl(2) -Te(1) -C(10) | 94.3 ( 1) | C(1) -C(6) -C(5) | 118.1 ( 4) |
| C(1) -Te(1) -C(10) | 97.8 ( 2) | C(1) -C(6) -C(7) | 123.5 ( 4) |
| C(7) -N(1) -C(8) | 110.6 ( 4) | C(5) -C(6) -C(7) | 118.2 ( 4) |
| C(7) -N(1) -C(9) | 112.1 ( 4) | N(1) -C(7) -C(6) | 111.8 ( 4) |
| C(8) -N(1) -C(9) | 111.0 ( 4) | Te(1) -C(10) -C(11) | 118.6 ( 4) |
| Te(1) -C(1) -C(2) | 118.9 ( 3) | C(10) -C(11) -C(12) | 110.3 ( 4) |
| Te(1) -C(1) -C(6) | 120.8 ( 3) | C(11) -C(12) -C(13) | 113.3 ( 5) |
| C(2) -C(1) -C(6) | 120.3 ( 4) | Cl(1) -Te(1) .Cl(3) | 97.5 ( 1) |
| Cl(2) -Te(1) .Cl(3) | 90.3 ('1) | C(1) -Te(1) .Cl(3) | 173.3 ( 2) |
| C(10) -Te(1) .Cl(3) | 76.8 ( 1) | | |

**TABLE 3.18**

**Mean Plane Analysis for**

**2-Dichloro(butyl)telluro-N-dimethylbenzylammonium Chloride**

Phenyl Ring

| Perpendicular Distance to Plane | |
|---|---|
| -0.00661 | C1 |
| 0.00342 | C2 |
| 0.00044 | C3 |
| -0.00103 | C4 |
| -0.00221 | C5 |
| 0.00599 | C6 |

Mean Dev = 0.003284. RMS Dev = 0.004030

Butyl Chain

| Perpendicular Distance to Plane | |
|---|---|
| -0.02809 | C10 |
| 0.02647 | C11 |
| 0.03025 | C12 |
| -0.02864 | C13 |

Mean Dev = 0.028361 RMS Dev = 0.028393

Figure 3.11. The structure of 2-dichloro(butyl)telluro-N-dimethylbenzylammonium chloride showing the atom numbering.

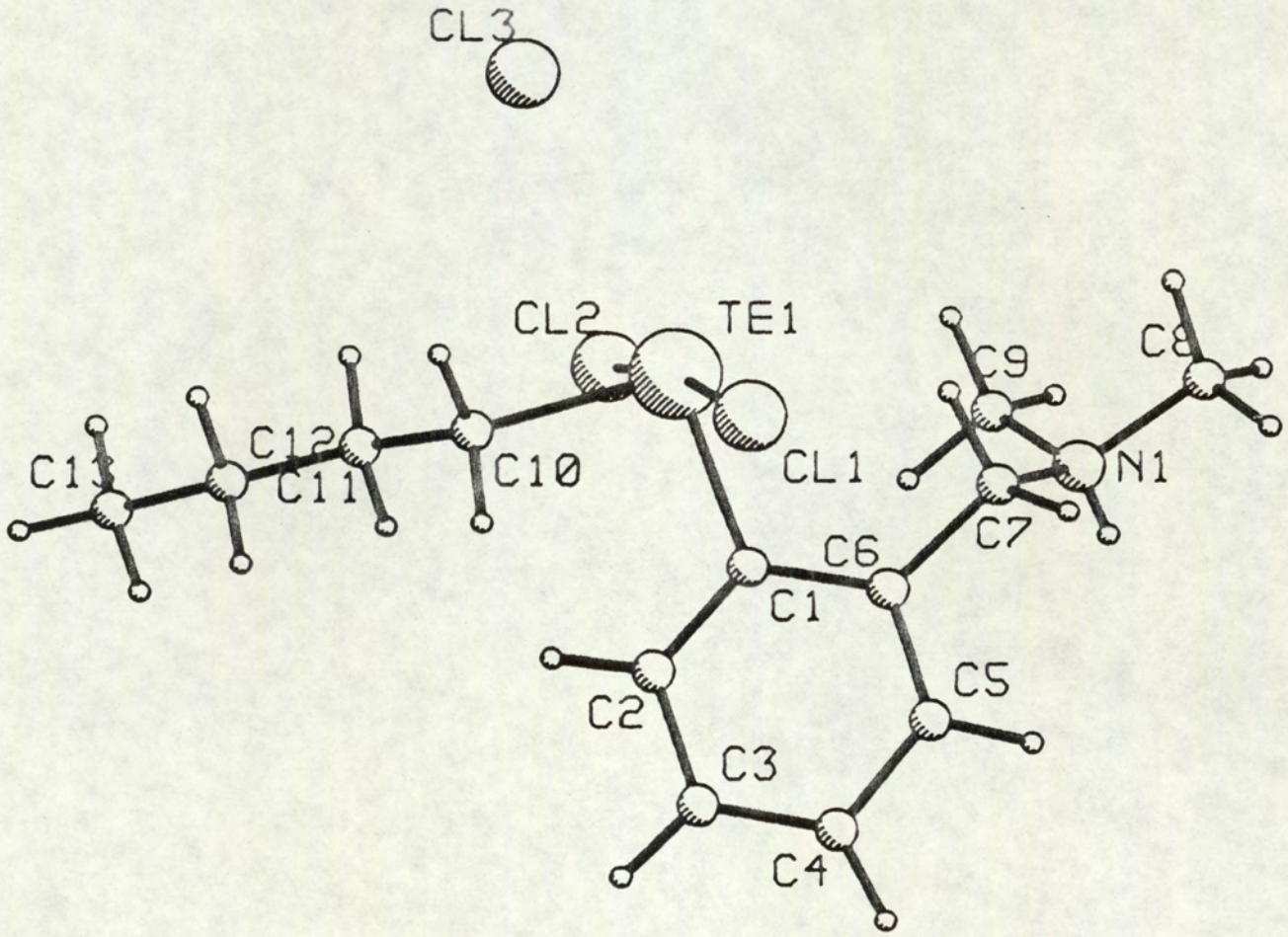
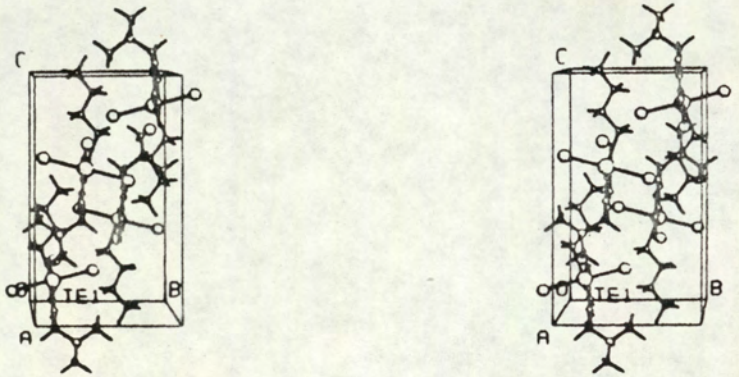


Figure 3.12. Stereoscopic packing diagram of 2-dichloro(butyl)telluro-N-dimethylbenzylammonium chloride





## 3.3 Tellurium (II) Structures

### 3.3.1 Dimethyldithiocarbamato[2-(2-pyridyl)phenyl] -tellurium(II), (7)

In the bulk of previous studies dealing with RTeX compounds, X has tended to be a halogen. In such instances the stability of the system has been due to complex formation brought about either by a reaction with a Lewis base or by intermolecular auto-complexation. The analysis of dimethyldithiocarbamato-[2-(2-pyridyl)phenyl]tellurium(II) provided an opportunity to study the effect of incorporating an electron donor in the organic group in an RTeX system. From the results of previous work, it was anticipated that this incorporation would stabilise the system by intramolecular autocomplex formation through a strong secondary tellurium nitrogen interaction. Broadening the underlying themes of this research of secondary tellurium nitrogen interactions and Lewis acidity of tellurium in varying environments into mono-organyl compounds of tellurium(II).

Bright yellow needle-like crystals were supplied by N Al-Salim of this department for analysis. The size of the crystal chosen for analysis was 0.60 x 0.25 x 0.08 mm.

**Crystal Data**

$C_{14}H_{14}N_2S_2Te$, Mr = 402.0, triclinic space group $P\bar{1}$, a = 8.809(3) Å, b = 9.032(5) Å, c = 10.727(4) Å, $\alpha$ = 83.06(4)°, $\beta$ = 86.49(3)°, $\gamma$ = 63.68(4)°, U = 759.4 $Å^3$, Z = 2, $D_c$ = 1.759 $gcm^{-3}$,

F(000) = 392, $\lambda$(Mo-$K_\alpha$) = 0.71069 Å, $\mu$ = 2.282 $mm^{-1}$. Unique data measured 2940 reflections, significant data [$I>2.5\theta(I)$] 2502 reflections.

With the exception of the ring hydrogens, which were located from a different synthesis, hydrogen atoms were placed in calculated positions (C-H 1.08 Å) and allowed to 'ride' on their respective carbon atoms in the subsequent least-squares refinements. Computer analysis was terminated when all shifts were less than $0.01\sigma$ and R and $R_w$ were 4.17% and 6.36% respectively; $w=1/(\sigma^2(F)+0.0025F^2)$.

Final atomic coordinates for dimethyldithiocarbamato[2-(2-pyridyl)phenyl]tellurium(II) are presented in table 3.19, with bond distances, bond angles and mean plane calculations given in tables 3.20 and 3.21 respectively. A view of the molecule and a stereoscopic packing diagram are shown in figures 3.13 and 3.14.

In dimethyldithiocarbamato[2-(2-pyridyl)phenyl]-tellurium(II) the basic molecular structure around tellurium can be considered as essentially $\psi$ trigonal bipyramidal. In this interpretation of the coordination around tellurium, the pyridyl nitrogen and the dimethyldithiocarbamate sulphur [S(1)] occupy the axial positions in the coordination sphere and the phenyl carbon [C(1)] along with the tellurium two lone pairs of electrons define the equatorial plane. The position *trans* to the phenyl carbon [C(1)] is thus unoccupied and the basic structure of the

molecule can be envisaged as "T" shaped[40]. Constraints imposed on the molecule by the five membered chelate ring [Te(1), C(1), C(8), C(7), N(1)], have distorted the coordination geometry away from ideal ψ trigonal bipyramidal values. This is particularly noticeable for the N(1)-Te(1)-C(1) angle which is contrained to a value of 74.4(2)° as opposed to an ideal value of 90°.

Focussing on the individual bond lengths involving tellurium and on the planarity of the ligands to which tellurium is attached. The tellurium $sp^2$ hybridised carbon distance of 2.111(5) Å is in good agreement with the sum of the Pauling single bond covalent radii (2.11 Å)[40,108] and with a number of values in other similar Te(II) complexes[62,63,104].

The tellurium nitrogen distance [2.354(4) Å] is greater than the combined values for an axial tellurium(II) nitrogen single covalent bond [2.23 Å][40,108]. This distance is also longer than that reported for the same interaction in $Te(C_6H_4N_2Ph)(SCN)$ (2.243 Å)[105] and $Te(C_6H_4N_2Ph)Cl$ (2.230Å)[102], but is never-the-less analogous to that recorded for $Te(C_6H_4N_2Ph)$dmdtc (2.340 Å)[104]. Interestingly, where there is a strong interaction between tellurium and nitrogen as apparently is the case here, the 2-(2-pyridyl)phenyl ligand is held in an almost planar geometry. In this instance, the ligand is planar to within ±0.06Å with only a 4.4(2)° angle between the mean planes of the pyridyl and phenyl rings. Situated 0.03Å from this plane is the tellurium atom, with the dimethyldithiocarbamate sulphur S(1) displaced by 0.30Å [table 3.21].

The Te-S(1) distance [2.518(1) Å] is comparable with the

theoretical value for an axial tellurium sulphur single covalent bond (2.58 Å) [40,108] and is similar to distances found in a range of other tellurium(II) complexes containing sulphur ligands (63,64,66,104).

There is a second tellurium sulphur contact within the molecule at a distance of 3.667(1) Å between tellurium and the second dimethyldithiocarbamate sulphur S(2). This distance, although within the sum of the van der Waals radii [4.05[108] or 3.91[107] Å] is twisted away from the fourth site to give a square planar geometry around tellurium, indicating that there is no significant interaction between the two atoms. In fact although the dimethyldithiocarbamate group is orientated at an angle of 72.7(6)° to the tellurium-organic ligand plane, the dimethyldithiocarbamate group is planar to within ±0.04Å.

Contrasting with this situation, the two sulphur atoms in dimethyldithiocarbamato(2-phenylazophenyl-C,N')tellurium(II) are essentially coplanar with the tellurium atom and the coordinating C and N atoms of the organic ligand, which results in a square planar geometry around tellurium. In this instance the Te-S(2) distance at 3.225(3) Å is considered to indicate a weak bond[104]. Within the unit cell no unusually short intermolecular contracts occur.

Table 3.19. Fractional atomic coordinates ($x10^5$ for tellurium and $x10^4$ for all other non-hyrogen atoms) with e.s.d.'s in parentheses for dimethyldithiocarbamato-2-(2-pyridyl)phenyltellurium(II) .

| | x | y | z |
|---|---|---|---|
| Te | 105314( 3) | 13542( 3) | 33091( 3) |
| S(1) | 9013( 2) | 1518( 2) | 1346( 1) |
| S(2) | 5995( 2) | 3887( 2) | 2727( 1) |
| N(1) | 12174( 5) | 1562( 5) | 4869( 4) |
| N(2) | 5861( 5) | 2549( 6) | 713( 4) |
| C(1) | 10496( 5) | 3713( 5) | 2887( 4) |
| C(2) | 9645( 6) | 4791( 6) | 1842( 5) |
| C(3) | 9632( 7) | 6328( 7) | 1632( 6) |
| C(4) | 10470( 7) | 6795( 6) | 2417( 6) |
| C(5) | 11331( 7) | 5731( 6) | 3463( 5) |
| C(6) | 11368( 6) | 4187( 5) | 3690( 4) |
| C(7) | 12295( 6) | 2992( 6) | 4743( 4) |
| C(8) | 13305( 8) | 3254( 7) | 5579( 5) |
| C(9) | 14156( 9) | 2030(10) | 6489( 6) |
| C(10) | 14017( 7) | 540( 8) | 6609( 5) |
| C(11) | 13012( 7) | 361( 7) | 5768( 5) |
| C(12) | 6811( 6) | 2680( 5) | 1556( 4) |
| C(13) | 3998( 8) | 3513( 9) | 714( 6) |
| C(14) | 6548( 9) | 1467(10) | -296( 6) |

Table 3.20 Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses for dimethyldithiocarbamato-2-(2-pyridyl)phenyltellurium(II)

| | | | | | |
|---|---|---|---|---|---|
| Te | ---S(1) | 2.518 ( 1) | C(1) | ---C(6) | 1.406 ( 7) |
| Te | ---N(1) | 2.354 ( 4) | C(2) | ---C(3) | 1.375 ( 8) |
| Te | ---C(1) | 2.111 ( 5) | C(3) | ---C(4) | 1.367 ( 9) |
| S(1) | ---C(12) | 1.764 ( 4) | C(4) | ---C(5) | 1.398 ( 8) |
| S(2) | ---C(12) | 1.676 ( 5) | C(5) | ---C(6) | 1.373 ( 7) |
| N(1) | ---C(7) | 1.332 ( 7) | C(6) | ---C(7) | 1.464 ( 6) |
| N(1) | ---C(11) | 1.333 ( 7) | C(7) | ---C(8) | 1.410 ( 7) |
| N(2) | ---C(12) | 1.319 ( 6) | C(8) | ---C(9) | 1.353 ( 9) |
| N(2) | ---C(13) | 1.478 ( 7) | C(9) | ---C(10) | 1.395 (10) |
| N(2) | ---C(14) | 1.464 ( 7) | C(10) | ---C(11) | 1.372 ( 8) |
| C(1) | ---C(2) | 1.400 ( 6) | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| S(1) | -Te | -N(1) | 167.6 ( 1) | C(3) | -C(4) | -C(5) | 120.7 ( 5) |
| S(1) | -Te | -C(1) | 95.0 ( 1) | C(4) | -C(5) | -C(6) | 119.7 ( 5) |
| N(1) | -Te | -C(1) | 74.4 ( 2) | C(1) | -C(6) | -C(5) | 119.5 ( 5) |
| Te | -S(1) | -C(12) | 109.6 ( 1) | C(1) | -C(6) | -C(7) | 118.1 ( 4) |
| Te | -N(1) | -C(7) | 113.8 ( 3) | C(5) | -C(6) | -C(7) | 122.4 ( 5) |
| Te | -N(1) | -C(11) | 125.2 ( 4) | N(1) | -C(7) | -C(6) | 115.8 ( 4) |
| C(7) | -N(1) | -C(11) | 120.9 ( 5) | N(1) | -C(7) | -C(8) | 119.8 ( 5) |
| C(12) | -N(2) | -C(13) | 121.8 ( 5) | C(6) | -C(7) | -C(8) | 124.4 ( 5) |
| C(12) | -N(2) | -C(14) | 123.4 ( 5) | C(7) | -C(8) | -C(9) | 119.0 ( 5) |
| C(13) | -N(2) | -C(14) | 114.8 ( 4) | C(8) | -C(9) | -C(10) | 120.5 ( 5) |
| Te | -C(1) | -C(2) | 122.0 ( 4) | C(9) | -C(10) | -C(11) | 117.7 ( 5) |
| Te | -C(1) | -C(6) | 118.0 ( 3) | N(1) | -C(11) | -C(10) | 122.0 ( 5) |
| C(2) | -C(1) | -C(6) | 120.1 ( 4) | S(1) | -C(12) | -S(2) | 122.3 ( 3) |
| C(1) | -C(2) | -C(3) | 119.3 ( 5) | S(1) | -C(12) | -N(2) | 115.1 ( 4) |
| C(2) | -C(3) | -C(4) | 120.7 ( 5) | S(2) | -C(12) | -N(2) | 122.7 ( 4) |

**TABLE 3.21**

**Mean Plane Analysis for**

**Dimethyldithiocarbamato[2-(2-pyridyl)phenyl]tellurium(II)**

2-(2-pyridyl)phenyl ligand.

Perpendicular Distance to Plane

| | |
|---|---|
| 0.05450 | N1 |
| -0.00073 | C1 |
| -0.05529 | C2 |
| -0.03706 | C3 |
| 0.00459 | C4 |
| 0.05445 | C5 |
| 0.03273 | C6 |
| 0.03616 | C7 |
| -0.01098 | C8 |
| -0.05557 | C9 |
| -0.03560 | C10 |
| 0.01281 | C11 |

Mean Dev=0.032538 RMS Dev=0.038085

The atoms Te(1) and S(1) lie perpendicular to this plane at 0.02553 and -0.29764 Ås respectively.

Phenyl Ring

| Perp. Dist. to Plane | |
|---|---|
| 0.00810 | C1 |
| -0.00578 | C2 |
| 0.00334 | C3 |
| -0.00321 | C4 |
| 0.00559 | C5 |
| -0.00804 | C6 |

Mean Dev=0.005679 RMS Dev=0.006007

Pyridyl Ring

| Perp. Dist. to Plane | |
|---|---|
| 0.00237 | N1 |
| -0.00305 | C7 |
| 0.00357 | C8 |
| -0.00335 | C9 |
| 0.00252 | C10 |
| -0.00206 | C11 |

Mean Dev=0.002821 RMS Dev=0.002873

Angle between the mean planes of the phenyl and pyridyl ring is 4.4(3)°.

Dithiocarbamate Ligand

| Perpendicular Distance to Plane | |
|---|---|
| -0.03220 | S1 |
| 0.02279 | S2 |
| 0.00573 | N2 |
| 0.00932 | C12 |
| -0.03654 | C13 |
| 0.03090 | C14 |

Mean Dev = 0.022911 RMS Dev = 0.025708

The angle between the mean planes of the dimethyldithiocarbamate ligand and the 2-(2-pyridyl)phenyl ligand is 72.7(3)°.

Figure 3.13. The structure of dimethyldithiocarbamato-2-(2-pyridyl)phenyltellurium(II) showing the atom numdering.

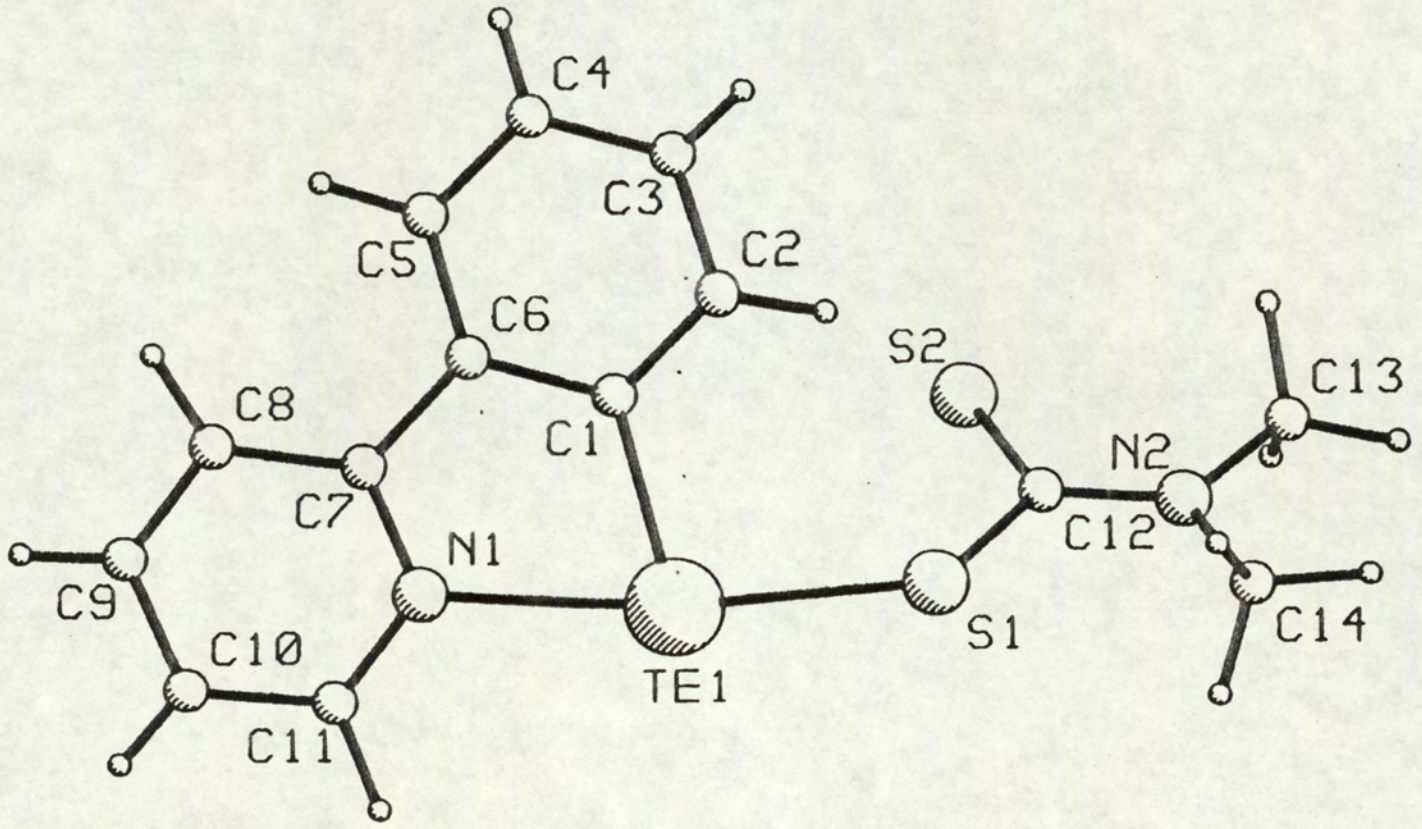
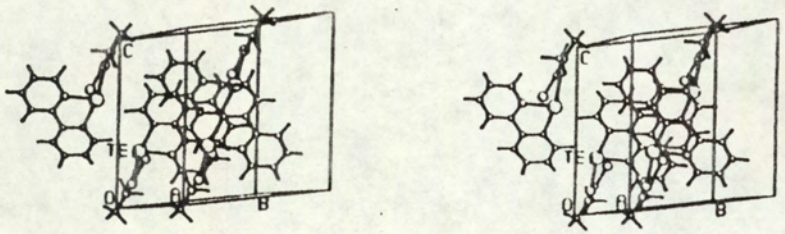


Figure 3.14. Stereoscopic packing diagram of dimethyldithiocarbamato-2-(2-pyridyl)phenyltellurium(II)





### 3.3.2 Dimethyldithiocarbamato[2-(2-quinolinyl) phenyltellurium(II), (8)

The analysis of dimethyldithiocarbamato[2-(2-quinolinyl) - phenyl]tellurium(II) although of interest in its own right, was primarily undertaken as a follow up to the earlier structural study of dimethyldithiocarbamato[2-(2-pyridyl)phenyltellurium(II). In that analysis, a short Te-N interaction occurred between tellurium and an electron donating nitrogen contained within the pyridyl ring of the 2-(2-pyridyl)phenyl ligand. The study of dimethyldithiocarbamato[2-(2-quinolinyl)phenyl]tellurium(II) therefore facilitated a second chance to examine the effect of incorporating an electron donating nitrogen into the organic ligand of an RTeX compound, as well as providing an opportunity to examine the effect of the ligand type *trans* to the nitrogen on the supposed Te-N interaction.

Yellow/orange crystals were supplied for analysis by N Al-Salim of this department. The size of the single crystal picked for the structural study was 0.60 x 0.30 x 0.25 mm.

Elemental Analysis: Theoretical values (%age) C 47.8 H 3.57 N 6.20 S 14.20. Analytical Results (%age) C 48.07 H 3.59 N 6.15, S 14.70.

**Crystal Data**

$C_{18}H_{16}N_2S_2Te$, Mr = 452.1, triclinic space group $P\bar{1}$, a = 7.360(5) Å, b = 15.022(10) Å, c = 17.730(10) Å, $\alpha$ = 114.26(2)°, ß = 94.12°, $\gamma$ = 95.63(3)°, U = 1764.9 Å$^3$, Z = 4, $D_c$ = 1.701 gcm$^{-3}$, F(000) = 888, $\lambda$(Mo-$K_\alpha$) = 0.71069 Å, $\mu$ = 1.97 mm$^{-1}$.

Total data measured 6400 reflections; significant data [$I>2.5\theta(I)$] 4969 reflections. Least squares refinement was ceased when all shifts were less than $0.1\sigma$ and the refinements had converged to R = 0.0336 and Rw = 0.0485, using a weighting scheme of $w=1/[\sigma^2(F)+0.0006F^2]$.

Final atomic coordinates for dimethyldithiocarbamato-[2-(2-quinolinyl)phenyl]tellurium(II) are given in table 3.22, with bond distances, bond angles and mean plane calculations given in tables 3.23 and 3.24 respectively. A view of the molecule and a stereocopic packing diagram are shown in figures 3.15 and 3.16.

Within the asymmetric unit are two crystallographically independent molecules, which have an essentially $\psi$ trigonal bipyramidal arrangement of atoms around tellurium. (The geometries of the two distinct molecules are quite similar, therefore only molecule A is shown in Figure 3.15). In each molecule the coordination sphere around tellurium is outlined by the pyridyl nitrogen and the dimethyldithiocarbamate sulphur S(1), which occupy the axial positions and by the 2-(2-quinolinyl)phenyl carbon C(1) and the two lone pairs of electrons on tellurium which define the equatorial plane. In this representation of the environment around tellurium, the position *trans* to the bonded phenyl carbon is unoccupied and the compound can be regarded as almost "T-shaped"[40]. An alternative view of the arrangement of atoms around tellurium, may be gained by including the second dimethyldithiocarbamate sulphur, S(2) into the coordination sphere. This approach by S(2) to tellurium [Te(1)...S(2) 3.222(1) and 3.230(1) Å] bisects the angle between the two tellurium lone

pairs of electrons and if considered to be significant results in an environment about tellurium which could be judged to be a manifestation of the tendency for $Te^{II}$ to achieve a square planar geometry.

Assuming the geometry around tellurium to be primarily $\psi$ trigonal bipyramidal, then the distortion of the structure away from ideal values can be explained in terms of the constraints imposed on the system by the five membered chelate ring [Te-N(1)-C(7)-C(6)-C(1)]. These constraints particularly affect the angle N(1)-Te-C(1), which is reduced to an angle of 74.5(1)° in molecule A and to an angle of 74.0(1)° in molecule B. This phenomenon is common to both this structure and to the 2-(2-pyridyl)phenyl structures which contain an identical five membered chelate ring system. The 2-(2-quinolinyl)phenyl ligand is planar to within ±0.15 Å in molecule A and to within ± 0.10 Å in molecule B, with a twist of 8.3(3)°, 5.3(2)° between the mean planes of the quinolinyl and phenyl rings. In the molecule, the tellurium atom is displaced from the 2-(2-quinolinyl)phenyl plane by 0.33 Å in molecule A and by 0.38 Å in molecule B, with the sulphur atoms S(1) and S(2) respectively removed by 0.94 Å and 0.78 Å and by 0.80 Å and 1.07 Å in molecules A and B. The dimethyldithiocarbamate group is essentially planar in both molecules and is orientated at an angle of 12.0(4)°, 3.9(2)° to the chelate ring plane in molecules A and B respectively.

The Te-S(1) bond length (2.569(1) Å, 2.543(1) Å) lies within the range of values reported for analogous tellurium(II) complexes containing sulphur ligands, for example,

$Te(C_6H_4N_2Ph)(dmdtc)$[104], $Te(Et_2NCS_2)_2$[72] and [TePh(etu)Br][64], range 2.52-2.57 Å and is in good agreement with the sum of the sulphur and axial tellurium single bond covalent radii (2.58 Å)[40,108]. The Te-S(2) distance (3.222(1) Å, 3,230(1) Å) is far greater than the sum of the Pauling single bond covalent radii for the two atoms [108]. This distance does however fall within the sum of the van der Waals radii (4.05 Å)[108] or (3.86 Å)[107] for the two atoms, which would tend to suggest that there is a weak secondary interaction taking place. This type of secondary interaction has been noted previously in $Te(C_6H_4N_2Ph)(dmdtc)$, where the contact distance between tellurium and sulphur was 3.225 Å[104]. In each molecule of dimethyldithiocarbamato-[2-(2-quinolinyl)]phenyltellurium(II), the two sulphur atoms are essentially coplanar with the tellurium atom and the coordinating carbon and nitrogen atoms of the organic ligand. This feature is akin to that found in dimethyldithiocarbamato-(2-phenylazophenyl-C,N') tellurium(II). Interestingly, however, these last two stuctural features are in contrast to that found in dimethyldithiocarbamato[2-(2-pyridyl)phenyl]tellurium(II), where the dimethyldithiocarbamate group is orientated at an angle of 72.7(3)° to the tellurium-organic ligand plane and the Te...S(2) distance at 3.667(1) Å was considered not to be a significant interaction.

The distance Te-C(1), (2.124(5), 2.135(4) Å) is in good agreement with the sum of the Pauling single bond covalent radii[108] and with values found in other tellurium(II) complexes

containing sulphur ligands [63,64,67,104], which lie in the range 2.101-2.124 Å.

The Te-N interaction (2.365(4) Å, 2.385(4) Å) exceeds the sum of the atoms single covalent bond radii and is also longer than the distances reported for the same interaction in $Te(C_6H_4N_2Ph)(SCN)$ (2.243 Å)[105] and $Te(C_6H_4N_2Ph)Cl$ (2.230 Å)[102]. This length is however similar to the values recorded for both $Te(C_6H_4N_2Ph)(dmdtc)$ (2.340 Å) [104] and $Te(C_5H_4NPh)(dmdtc)$. (2.354 Å), studied here. The planarity of the organic ligand which is commented on above, appears to be due to the Te-N interaction holding the ligand in a rigid geometry so as to prevent the rings of the ligand from twisting about the C(6)-C(7) bond. This aspect of the structure mimics the situation identified in the tellurium 2-(2-pyridyl)phenyl structures when there is a strong secondary interaction between tellurium and nitrogen. There are no other unusually short interactions within the unit cell.

Table 3.22. Fractional atomic coordinates ($x10^5$ for tellurium and $x10^4$ for all other non-hydrogen atoms) with e.s.d.'s in parentheses for dimethyldithiocarbamato-2-(2-quinolinyl)phenyltellurium(II).

| | x | y | z |
|---|---|---|---|
| Te(1A) | 29856( 3) | 73451( 2) | 222( 2) |
| S(1A) | 3022( 2) | 9211( 1) | 507( 1) |
| S(2A) | 2202( 2) | 8190( 1) | -1354( 1) |
| N(1A) | 2858( 4) | 5744( 2) | -24( 2) |
| N(2A) | 2200( 5) | 10102( 2) | -448( 2) |
| C(1A) | 3740( 5) | 7560( 3) | 1271( 3) |
| C(2A) | 4352( 6) | 8476( 3) | 1926( 3) |
| C(3A) | 4865( 7) | 8556( 4) | 2721( 3) |
| C(4A) | 4735( 7) | 7730( 5) | 2884( 3) |
| C(5A) | 4125( 7) | 6813( 4) | 2245( 3) |
| C(6A) | 3641( 5) | 6713( 3) | 1428( 3) |
| C(7A) | 3074( 5) | 5747( 3) | 729( 3) |
| C(8A) | 2732( 7) | 4844( 4) | 813( 3) |
| C(9A) | 2150( 6) | 4003( 4) | 145( 3) |
| C(10A) | 1897( 5) | 3977( 3) | -667( 3) |
| C(11A) | 2302( 5) | 4891( 3) | -722( 3) |
| C(12A) | 2151( 7) | 4898( 3) | -1517( 3) |
| C(13A) | 1547( 8) | 4042( 3) | -2222( 3) |
| C(14A) | 1078( 7) | 3164( 4) | -2162( 4) |
| C(15A) | 1273( 7) | 3123( 3) | -1407( 4) |
| C(16A) | 2452( 5) | 9221( 3) | -465( 3) |
| C(17A) | 1825( 7) | 10215( 4) | -1225( 3) |
| C(18A) | 2392( 6) | 11002( 3) | 319( 3) |

Table Continued. Molecule B

| | | | |
|---|---|---|---|
| Te(1B) | 28215( 3) | 72503( 2) | 50311( 2) |
| S(1B) | 1843( 2) | 8876( 1) | 5985( 1) |
| S(2B) | -882( 2) | 7902( 1) | 4443( 1) |
| N(1B) | 4565( 5) | 5930( 3) | 4422( 2) |
| N(2B) | -1156( 5) | 9572( 2) | 5738( 2) |
| C(1B) | 5187( 6) | 7549( 3) | 5913( 3) |
| C(2B) | 5444( 7) | 8327( 4) | 6694( 3) |
| C(3B) | 7043( 8) | 8525( 4) | 7220( 4) |
| C(4B) | 8395( 9) | 7945( 5) | 6965( 4) |
| C(5B) | 8182( 7) | 7156( 4) | 6200( 4) |
| C(6B) | 6532( 6) | 6940( 4) | 5663( 3) |
| C(7B) | 6204( 6) | 6086( 3) | 4845( 3) |
| C(8B) | 7529( 8) | 5427( 4) | 4501( 4) |
| C(9B) | 7076( 9) | 4660( 5) | 3775( 5) |
| C(10B) | 5288( 8) | 4466( 4) | 3294( 4) |
| C(11B) | 4050( 7) | 5142( 3) | 3662( 3) |
| C(12B) | 2274( 7) | 4985( 3) | 3233( 3) |
| C(13B) | 1722( 9) | 4191( 3) | 2487( 3) |
| C(14B) | 2986(11) | 3525( 4) | 2135( 4) |
| C(15B) | 4709(10) | 3665( 4) | 2523( 4) |
| C(16B) | -199( 6) | 8824( 3) | 5387( 3) |
| C(17B) | -2876( 7) | 9632( 4) | 5307( 4) |
| C(18B) | -576( 7) | 10369( 3) | 6577( 3) |

Table 3.23 Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses for dimethyldithiocarbamato-2-(2-quinolinyl)phenyl-tellurium(II)

| | | | |
|---|---|---|---|
| Te(1A)---S(1A) | 2.569 ( 1) | Te(1B)---S(1B) | 2.543 ( 1) |
| Te(1A)...S(2A) | 3.222 ( 1) | Te(1B)...S(2B) | 3.230 ( 1) |
| Te(1A)---N(1A) | 2.365 ( 4) | Te(1B)---N(1B) | 2.385 ( 4) |
| Te(1A)---C(1A) | 2.124 ( 5) | Te(1B)---C(1B) | 2.135 ( 4) |
| S(1A) ---C(16A) | 1.751 ( 5) | S(1B) ---C(16B) | 1.752 ( 5) |
| S(2A) ---C(16A) | 1.677 ( 4) | S(2B) ---C(16B) | 1.676 ( 4) |
| N(1A) ---C(7A) | 1.331 ( 7) | N(1B) ---C(7B) | 1.320 ( 6) |
| N(1A) ---C(11A) | 1.365 ( 4) | N(1B) ---C(11B) | 1.373 ( 5) |
| N(2A) ---C(16A) | 1.343 ( 6) | N(2B) ---C(16B) | 1.338 ( 5) |
| N(2A) ---C(17A) | 1.465 ( 8) | N(2B) ---C(17B) | 1.461 ( 7) |
| N(2A) ---C(18A) | 1.456 ( 5) | N(2B) ---C(18B) | 1.473 ( 5) |
| C(1A) ---C(2A) | 1.390 ( 5) | C(1B) ---C(2B) | 1.381 ( 6) |
| C(1A) ---C(6A) | 1.407 ( 8) | C(1B) ---C(6B) | 1.386 ( 6) |
| C(2A) ---C(3A) | 1.385 ( 8) | C(2B) ---C(3B) | 1.375 ( 8) |
| C(3A) ---C(4A) | 1.381 (10) | C(3B) ---C(4B) | 1.366 ( 9) |
| C(4A) ---C(5A) | 1.381 ( 7) | C(4B) ---C(5B) | 1.371 ( 7) |
| C(5A) ---C(6A) | 1.409 ( 8) | C(5B) ---C(6B) | 1.411 ( 7) |
| C(6A) ---C(7A) | 1.465 ( 5) | C(6B) ---C(7B) | 1.472 ( 6) |
| C(7A) ---C(8A) | 1.424 ( 8) | C(7B) ---C(8B) | 1.443 ( 8) |
| C(8A) ---C(9A) | 1.331 ( 6) | C(8B) ---C(9B) | 1.320 ( 8) |
| C(9A) ---C(10A) | 1.421 ( 9) | C(9B) ---C(10B) | 1.449 ( 9) |
| C(10A)---C(11A) | 1.420 ( 7) | C(10B)---C(11B) | 1.418 ( 7) |
| C(10A)---C(15A) | 1.412 ( 6) | C(10B)---C(15B) | 1.402 ( 7) |
| C(11A)---C(12A) | 1.410 ( 8) | C(11B)---C(12B) | 1.411 ( 7) |
| C(12A)---C(13A) | 1.379 ( 6) | C(12B)---C(13B) | 1.367 ( 6) |
| C(13A)---C(14A) | 1.378 ( 8) | C(13B)---C(14B) | 1.417 ( 9) |
| C(14A)---C(15A) | 1.363 (10) | C(14B)---C(15B) | 1.352 (10) |

| | | | |
|---|---|---|---|
| S(1A) -Te(1A)-N(1A) | 163.7 ( 1) | S(1B) -Te(1B)-N(1B) | 162.8 ( 1) |
| S(1A) -Te(1A)-C(1A) | 90.3 ( 1) | S(1B) -Te(1B)-C(1B) | 89.0 ( 1) |
| N(1A) -Te(1A)-C(1A) | 74.5 ( 1) | N(1B) -Te(1B)-C(1B) | 74.0 ( 1) |
| Te(1A)-S(1A) -C(16A) | 98.6 ( 1) | Te(1B)-S(1B) -C(16B) | 99.1 ( 1) |
| Te(1A)-N(1A) -C(7A) | 113.1 ( 2) | Te(1B)-N(1B) -C(7B) | 112.7 ( 3) |
| Te(1A)-N(1A) -C(11A) | 124.7 ( 3) | Te(1B)-N(1B) -C(11B) | 124.7 ( 3) |
| C(7A) -N(1A) -C(11A) | 121.3 ( 4) | C(7B) -N(1B) -C(11B) | 122.1 ( 4) |
| C(16A)-N(2A) -C(17A) | 120.6 ( 3) | C(16B)-N(2B) -C(17B) | 121.2 ( 3) |
| C(16A)-N(2A) -C(18A) | 123.2 ( 4) | C(16B)-N(2B) -C(18B) | 122.8 ( 4) |
| C(17A)-N(2A) -C(18A) | 116.0 ( 4) | C(17B)-N(2B) -C(18B) | 116.0 ( 4) |
| Te(1A)-C(1A) -C(2A) | 124.1 ( 4) | Te(1B)-C(1B) -C(2B) | 123.1 ( 4) |
| Te(1A)-C(1A) -C(6A) | 117.0 ( 3) | Te(1B)-C(1B) -C(6B) | 117.1 ( 3) |
| C(2A) -C(1A) -C(6A) | 118.9 ( 5) | C(2B) -C(1B) -C(6B) | 119.8 ( 4) |
| C(1A) -C(2A) -C(3A) | 120.8 ( 5) | C(1B) -C(2B) -C(3B) | 120.9 ( 5) |
| C(2A) -C(3A) -C(4A) | 120.6 ( 4) | C(2B) -C(3B) -C(4B) | 119.4 ( 5) |
| C(3A) -C(4A) -C(5A) | 119.7 ( 6) | C(3B) -C(4B) -C(5B) | 121.5 ( 5) |
| C(4A) -C(5A) -C(6A) | 120.5 ( 6) | C(4B) -C(5B) -C(6B) | 119.3 ( 5) |
| C(1A) -C(6A) -C(5A) | 119.4 ( 4) | C(1B) -C(6B) -C(5B) | 119.0 ( 4) |
| C(1A) -C(6A) -C(7A) | 118.7 ( 5) | C(1B) -C(6B) -C(7B) | 119.3 ( 4) |
| C(5A) -C(6A) -C(7A) | 121.9 ( 5) | C(5B) -C(6B) -C(7B) | 121.7 ( 4) |
| N(1A) -C(7A) -C(6A) | 115.9 ( 5) | N(1B) -C(7B) -C(6B) | 115.9 ( 4) |
| N(1A) -C(7A) -C(8A) | 119.7 ( 4) | N(1B) -C(7B) -C(8B) | 119.8 ( 4) |
| C(6A) -C(7A) -C(8A) | 124.4 ( 5) | C(6B) -C(7B) -C(8B) | 124.3 ( 4) |
| C(7A) -C(8A) -C(9A) | 120.2 ( 6) | C(7B) -C(8B) -C(9B) | 119.6 ( 5) |
| C(8A) -C(9A) -C(10A) | 121.3 ( 6) | C(8B) -C(9B) -C(10B) | 122.0 ( 6) |
| C(9A) -C(10A)-C(11A) | 116.3 ( 4) | C(9B) -C(10B)-C(11B) | 115.8 ( 4) |
| C(9A) -C(10A)-C(15A) | 125.1 ( 5) | C(9B) -C(10B)-C(15B) | 125.1 ( 6) |
| C(11A)-C(10A)-C(15A) | 118.5 ( 5) | C(11B)-C(10B)-C(15B) | 119.1 ( 5) |
| N(1A) -C(11A)-C(10A) | 121.0 ( 5) | N(1B) -C(11B)-C(10B) | 120.7 ( 4) |
| N(1A) -C(11A)-C(12A) | 120.4 ( 4) | N(1B) -C(11B)-C(12B) | 120.8 ( 4) |
| C(10A)-C(11A)-C(12A) | 118.6 ( 4) | C(10B)-C(11B)-C(12B) | 118.5 ( 4) |

| | | | |
|---|---|---|---|
| C(11A)-C(12A)-C(13A) | 120.6 ( 5) | C(11B)-C(12B)-C(13B) | 121.6 ( 5) |
| C(12A)-C(13A)-C(14A) | 120.6 ( 6) | C(12B)-C(13B)-C(14B) | 118.7 ( 6) |
| C(13A)-C(14A)-C(15A) | 120.4 ( 4) | C(13B)-C(14B)-C(15B) | 121.2 ( 5) |
| C(10A)-C(15A)-C(14A) | 121.3 ( 5) | C(10B)-C(15B)-C(14B) | 120.8 ( 6) |
| S(1A) -C(16A)-S(2A) | 121.8 ( 3) | S(1B) -C(16B)-S(2B) | 121.9 ( 3) |
| S(1A) -C(16A)-N(2A) | 115.6 ( 3) | S(1B) -C(16B)-N(2B) | 115.2 ( 3) |
| S(2A) -C(16A)-N(2A) | 122.6 ( 4) | S(2B) -C(16B)-N(2B) | 122.9 ( 3) |

TABLE 3.24

**Mean Plane Analysis for**

**Dimethyldithiocarbamato[2-(2-quinolinylphenyl]tellurium](II)**

| Molecule A<br>2-(2-Quinolinyl)phenyl Ring<br>Perpendicular Distance to Plane | | Molecule B<br>2-(2-Quinolinyl)phenyl Ring<br>Perpendicular Distance to Plane | |
|---|---|---|---|
| -0.02624 | N1 | -0.05199 | N1 |
| 0.15139 | C1 | -0.09731 | C1 |
| 0.11905 | C2 | -0.02035 | C2 |
| -0.03644 | C3 | 0.07945 | C3 |
| -0.12769 | C4 | 0.09880 | C4 |
| -0.09424 | C5 | 0.04476 | C5 |
| 0.02902 | C6 | -0.03457 | C6 |
| 0.00355 | C7 | -0.05386 | C7 |
| 0.02486 | C8 | -0.05635 | C8 |
| 0.05263 | C9 | -0.02383 | C9 |
| 0.02892 | C10 | -0.01972 | C10 |
| -0.02837 | C11 | -0.02616 | C11 |
| -0.11271 | C12 | 0.00198 | C12 |
| -0.08247 | C13 | 0.06345 | C13 |
| 0.03271 | C14 | 0.07403 | C14 |
| 0.06603 | C15 | 0.02168 | C15 |
| Mean Dev=0.063519 | | MeanDev=0.048019 | |
| RMS Dev=0.077101 | | RMS Dev=0.055654 | |

The following atoms lie perpendicular to this plane at a distance of

| | | | |
|---|---|---|---|
| 0.33259 | Te1 | -0.37857 | Te1 |
| 0.93941 | S1 | -0.78211 | S1 |
| 0.79521 | S2 | -1.07250 | S2 |

Angle between the Mean Planes of the Phenyl and Quinolinyl rings, 8.3(3)$^{o}$ for Moleucle A and 5.3(2)$^{o}$ for Molecule B.

Dimethyldithiocarbamate Group

| Perpendicular Distance to Plane | | Perpendicular Distance to Plane | |
|---|---|---|---|
| -0.02528 | S1 | -0.01185 | S1 |
| 0.01960 | S2 | 0.01257 | S2 |
| 0.02248 | N2 | -0.00591 | N2 |
| -0.00121 | C16 | 0.00051 | C16 |
| -0.03496 | C17 | -0.01233 | C17 |
| 0.01936 | C18 | 0.01701 | C18 |
| Mean Dev=0.020482 | | Mean Dev=0.010030 | |
| RMS Dev=0.022830 | | RMS Dev=0.011366 | |

Angle between the Mean Planes of the 2-(2-quinolinyl)phenyl ring and the dimethyl -dithiocarbamate group is 17.9(3)° in Molecule A and 10.0(2)° in Molecule B.

Figure 3.15 The structure of dimethyldithiocarbamato-2-(2-quinolinyl)phenyltellurium(II) showing the atom numbering.

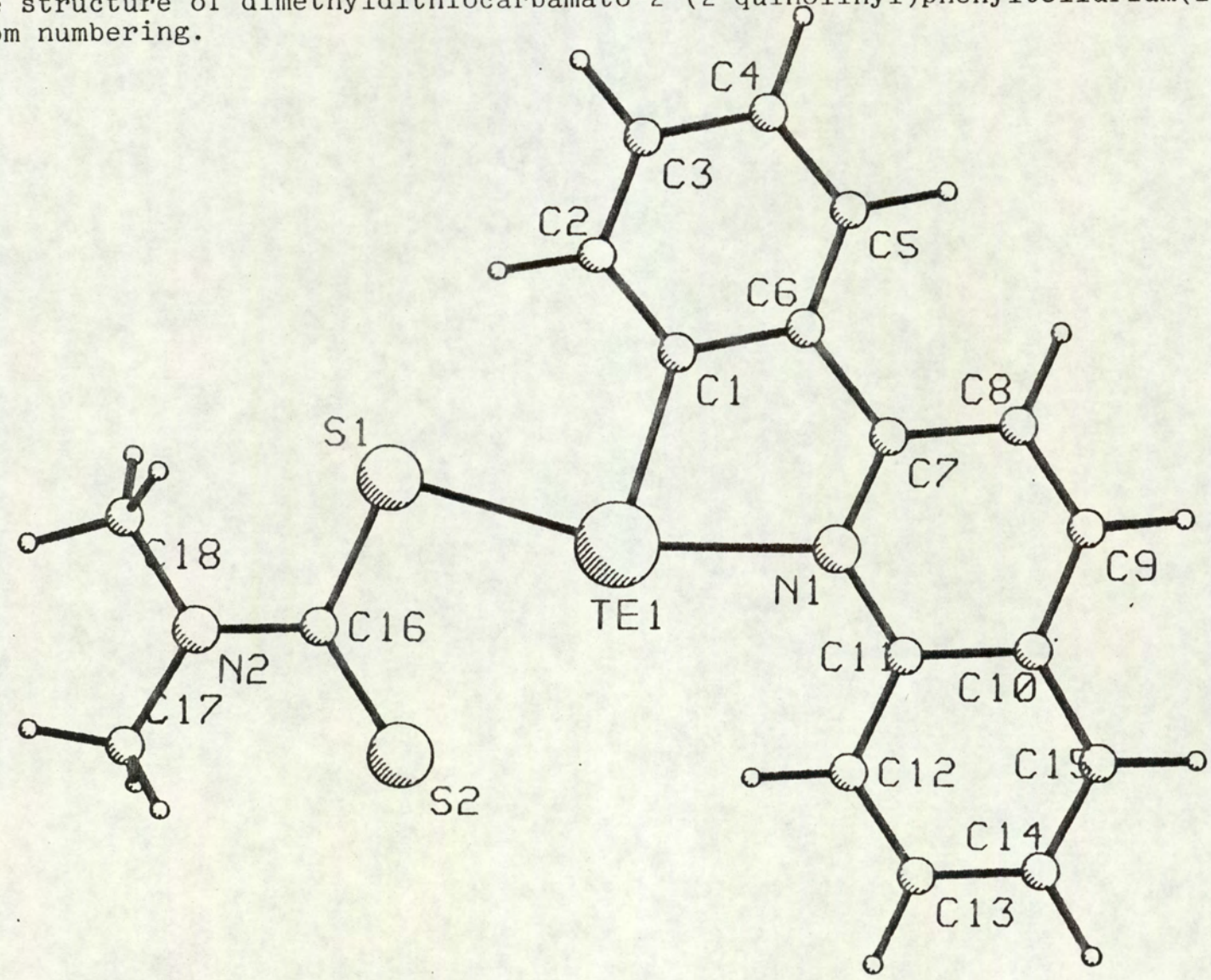
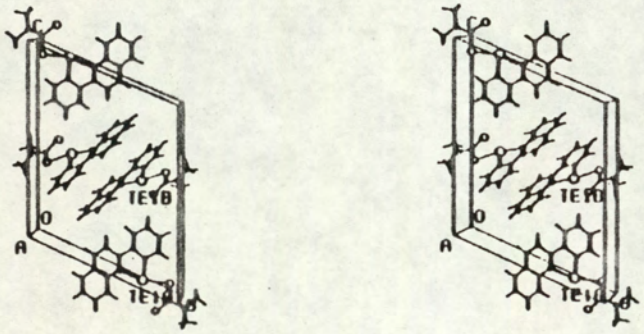


Figure 3.16. Stereoscopic packing diagram of dimethyldithiocarbamato-2-(2-quinolinyl)phenyltellurium(II)





### 3.3.3 *p*-Ethoxyphenyl[2-(2-pyridyl)phenyl]telluride, (9)

At first glance, the crystallographic study of *p*-Ethoxyphenyl [2-(2-pyridyl)phenyl]telluride may appear an unusual compound to include in an investigation examining the Lewis acidity of tellurium. This is because generally, diorganyltellurides show quite strong Lewis base behaviour. However, tempting as it may be to dismiss diorganyltellurides from this work, it is too sweeping to suggest that no Lewis acid behaviour has been observed[40]. The study of *p*-ethoxyphenyl[2-(2-pyridyl)phenyl]-telluride provided an opportunity of examining an $R_2Te$ system, with an electron donoting nitrogen contained within the pyridyl ring of the 2-(2-pyridyl)phenyl ligand. Interest in this study naturally centred on how the supposed Te-N interestion would be effected by the low Lewis acidity of tellurium and the anticipated fact that the interaction would lie *trans* to a Te-C bond.

Pale yellow crystals of *p*-ethoxyphenyl[2-(2-pyridyl)phenyl] -telluride were furnished for analysis by N. Al-Salim of this department. The dimensions of the needle like crystal selected for X-ray analysis were 0.55 x 0.25 x 0.125 mm.

**Crystal Data**

$C_{19}H_{17}NOTe$, Mr = 402.9 monoclinic space group $P2_1/C$, a = 13.422(2) Å, b = 16.469(3) Å, c = 7.711(3) Å, ß = 91.01(2)°, U = 1704.2 $Å^3$, Z = 4, $D_C$ = 1.571 $gcm^3$, F(000) = 792, $\lambda$(Mo-$K_\alpha$) = 0.71069 Å, $\mu$ = 1.813$mm^{-1}$.

3356 unique reflections were measured in a data range $2<\theta<22.5$, of these 2014 significant reflections with $I>2.5\theta(I)$ were used in the computational construction of the structure. Computer least squares analysis was terminated when all shifts were less than $0.01\sigma$ and R and $R_w$ were 0.0415 and 0.0539 respectively; $w = {}^1/[\sigma^2(F)+0.0015F^2]$

Final atomic coordinates for *p*-ethoxyphenyl [2-(2-pyridyl)phenyl]telluride are given in table 3.25, with bond distances, bond angles, torsion angles and mean plane calculation given in tables 3.26 and 3.27 respectively. A view of the molecule and a stereoscopic packing diagram are shown in figures 3.17 and 3.18.

The coordination sphere around tellurium is somewhat difficult to define, as this atom is bonded only primarily to two carbon atoms. A third weak contact occurs at 2.695(4) Å with the pyridyl ring nitrogen and if this interaction is considered to be significant then the coordination sphere around tellurium can best be visualised as trigonal bipyramidal. In this representation of the coordination sphere, the equatorial plane is defined by C(1) and the two lone pairs of electrons on tellurium and the axial positions are taken up by C(12) and N(1).

The Te-N contact distance at 2.695(4)Å although significantly longer than the same interaction in all the other structures studied during this work which contain the 2-(2-pyridyl)phenyl ligand, (range 2.244-2.554 Å) is similar in magnitude to the Te...N interaction in

1,6bis(2-butotellurophenyl)-2,5-diazahex-1,5-diene [2.773 Å] (106). Even allowing for the "poor" Lewis acidity of tellurium in this environment, the fact that a Te-C covalent bond is positioned *trans* to the Te...N vector apparently seems to weaken the interaction. The relative weakness of this contact appears to enable the pyridyl and phenyl rings to twist relative to eachother, giving a twist of 23.2(2)° between the mean planes of the two rings. This result is in contrast to that of comparable structures described previously, where the relatively strong Te...N interaction appeared to hold the 2-(2-pyridyl)phenyl ligand in an essentially planar geometry.

The two tellurium carbon distances, Te-C(1) [2.138(6) Å] and Te-C(12) [2.144(6) Å] are in good agreement with the sum of the Pauling single bond covalent radii(108) and with values reported for the crystal structures of analogous diorganyltellurium(II) compounds(161-166) which lie in the range 2.050-2.140 Å. The ethoxy and phenyl groups of the *p*-ethoxyphenyl ligand are each planar, with the ethoxy group oriented at an angle 4.3(1)° to the phenyl ring.

There is a short Te...Te contact of 3.908Å, within the unit cell. This contact, although short, is not regarded as an example of coordination, as such contacts are common in tellurides and are often significantly shorter than the van der Waals radii. Bearing this contact in mind there are no other unusually short intermolecular contacts within the structure.

Table 3.25 Fractional atomic coordinates ($x10^5$ for tellurium and $x10^4$ for all other non-hydrogen atoms ) with e.s.d.'s in parentheses for p-ethoxyphenyl[2-(2-pyridyl)phenyl]telluride.

| | x | y | z |
|---|---|---|---|
| Te | 12013( 3) | -2182( 3) | -13169( 5) |
| O(1) | 3726( 4) | 1292( 3) | 4833( 6) |
| N(1) | 445( 4) | -1079( 4) | -4005( 7) |
| C(1) | 2325( 4) | -1013( 4) | -2275( 8) |
| C(2) | 3301( 5) | -944( 4) | -1675( 8) |
| C(3) | 4041( 5) | -1425( 5) | -2353(10) |
| C(4) | 3803( 6) | -1975( 5) | -3648(10) |
| C(5) | 2858( 5) | -2068( 5) | -4222( 9) |
| C(6) | 2075( 5) | -1595( 4) | -3562( 8) |
| C(7) | 1049( 5) | -1707( 4) | -4209( 7) |
| C(8) | 708( 6) | -2422( 5) | -4983(10) |
| C(9) | -256( 6) | -2471( 6) | -5548(11) |
| C(10) | -880( 6) | -1825( 6) | -5362(10) |
| C(11) | -496( 6) | -1138( 5) | -4562(10) |
| C(12) | 2110( 5) | 246( 4) | 773( 8) |
| C(13) | 2428( 5) | 1037( 5) | 739( 9) |
| C(14) | 2966( 5) | 1380( 4) | 2097( 9) |
| C(15) | 3208( 5) | 911( 5) | 3525( 9) |
| C(16) | 2912( 6) | 114( 5) | 3606(10) |
| C(17) | 2357( 6) | -224( 5) | 2220( 9) |
| C(18) | 3971( 6) | 835( 6) | 6357(10) |
| C(19) | 4596( 8) | 1331( 6) | 7514(13) |

Table 3.26, Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses for p-ethoxyphenyl[2-(2-pyridyl)phenyl]telluride.

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Te | ---C(1) | 2.138 ( 6) | | C(6) | ---C(7) | 1.468 ( 9) | |
| Te | ---C(12) | 2.144 ( 6) | | C(7) | ---C(8) | 1.394 (10) | |
| O(1) | ---C(15) | 1.368 ( 8) | | C(8) | ---C(9) | 1.359 (11) | |
| O(1) | ---C(18) | 1.430 ( 9) | | C(9) | ---C(10) | 1.364 (12) | |
| N(1) | ---C(7) | 1.325 ( 8) | | C(10) | ---C(11) | 1.384 (11) | |
| N(1) | ---C(11) | 1.331 ( 9) | | C(12) | ---C(13) | 1.372 (10) | |
| C(1) | ---C(2) | 1.387 ( 9) | | C(12) | ---C(17) | 1.393 (10) | |
| C(1) | ---C(6) | 1.415 ( 9) | | C(13) | ---C(14) | 1.382 (10) | |
| C(2) | ---C(3) | 1.380 (10) | | C(14) | ---C(15) | 1.380 (10) | |
| C(3) | ---C(4) | 1.382 (11) | | C(15) | ---C(16) | 1.372 (10) | |
| C(4) | ---C(5) | 1.346 (10) | | C(16) | ---C(17) | 1.408 (10) | |
| C(5) | ---C(6) | 1.410 ( 9) | | C(18) | ---C(19) | 1.463 (12) | |
| Te(1) | ...N(1) | 2.695 ( 4) | | | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| C(1) | -Te | -C(12) | 94.8 ( 2) | C(7) | -C(8) | -C(9) | 119.5 ( 8) |
| C(15) | -O(1) | -C(18) | 118.1 ( 6) | C(8) | -C(9) | -C(10) | 120.2 ( 8) |
| C(7) | -N(1) | -C(11) | 118.9 ( 7) | C(9) | -C(10) | -C(11) | 117.4 ( 7) |
| Te | -C(1) | -C(2) | 120.2 ( 5) | N(1) | -C(11) | -C(10) | 123.2 ( 8) |
| Te | -C(1) | -C(6) | 119.8 ( 4) | Te | -C(12) | -C(13) | 119.8 ( 5) |
| C(2) | -C(1) | -C(6) | 120.0 ( 6) | Te | -C(12) | -C(17) | 121.9 ( 5) |
| C(1) | -C(2) | -C(3) | 120.6 ( 7) | C(13) | -C(12) | -C(17) | 118.3 ( 6) |
| C(2) | -C(3) | -C(4) | 119.4 ( 7) | C(12) | -C(13) | -C(14) | 122.2 ( 7) |
| C(3) | -C(4) | -C(5) | 121.1 ( 7) | C(13) | -C(14) | -C(15) | 119.2 ( 7) |
| C(4) | -C(5) | -C(6) | 121.5 ( 7) | O(1) | -C(15) | -C(14) | 116.2 ( 7) |
| C(1) | -C(6) | -C(5) | 117.3 ( 6) | O(1) | -C(15) | -C(16) | 123.3 ( 7) |
| C(1) | -C(6) | -C(7) | 122.2 ( 6) | C(14) | -C(15) | -C(16) | 120.5 ( 7) |
| C(5) | -C(6) | -C(7) | 120.5 ( 6) | C(15) | -C(16) | -C(17) | 119.6 ( 7) |
| N(1) | -C(7) | -C(6) | 115.7 ( 6) | C(12) | -C(17) | -C(16) | 120.2 ( 7) |
| N(1) | -C(7) | -C(8) | 120.8 ( 6) | O(1) | -C(18) | -C(19) | 109.2 ( 8) |
| C(6) | -C(7) | -C(8) | 123.5 ( 6) | | | | |

## TABLE 3.27

### Mean Plane Analysis for *p*-Ethoxyphenyl[2-(2-pyridyl)phenyl]telluride

| Phenyl Ring | | Pyridyl Ring | |
|---|---|---|---|
| Perpendicular Distance to Plane | | Perpendicualr Distance to Plane | |
| -0.01254 | C1 | -0.00438 | C7 |
| 0.00393 | C2 | 0.00208 | C8 |
| 0.00865 | C3 | 0.00322 | C9 |
| -0.01245 | C4 | -0.00639 | C10 |
| 0.00355 | C5 | 0.00438 | C11 |
| 0.00887 | C6 | 0.00109 | N1 |
| Mean Dev=0.008332 | | Mean Dev=0.003590 | |
| RMS Dev=0.009073 | | RMS Dev=0.003980 | |

Angle between the Mean Planes of the Phenyl and Pyridyl Rings 23.2(2)°.

*p*-Ethoxyphenyl ligand

| Perpendicular Distance to Plane | |
|---|---|
| 0.00320 | C12 |
| -0.00518 | C13 |
| 0.00102 | C14 |
| -0.00777 | C15 |
| -0.01208 | C16 |
| -0.00608 | C17 |
| 0.02243 | 01 |
| 0.04795 | C18 |
| -0.04349 | C19 |

Mean Dev=0.016577 RMS Dev=0.023511

Torsion Angles

| | |
|---|---|
| C(1)-C(6)-C(7)-C(8) | -157.1 |
| C(5)-C(6)-C(7)-C(8) | 23.3 |
| C(1)-C(6)-C(7)-N(1) | 22.5 |
| C(5)-C(6)-C(7)-N(1) | -157.1 |

Figure 3.17. The structure of p-ethoxyphenyl[2-(2-pyridyl)phenyl]telluride showing the atom numbering.

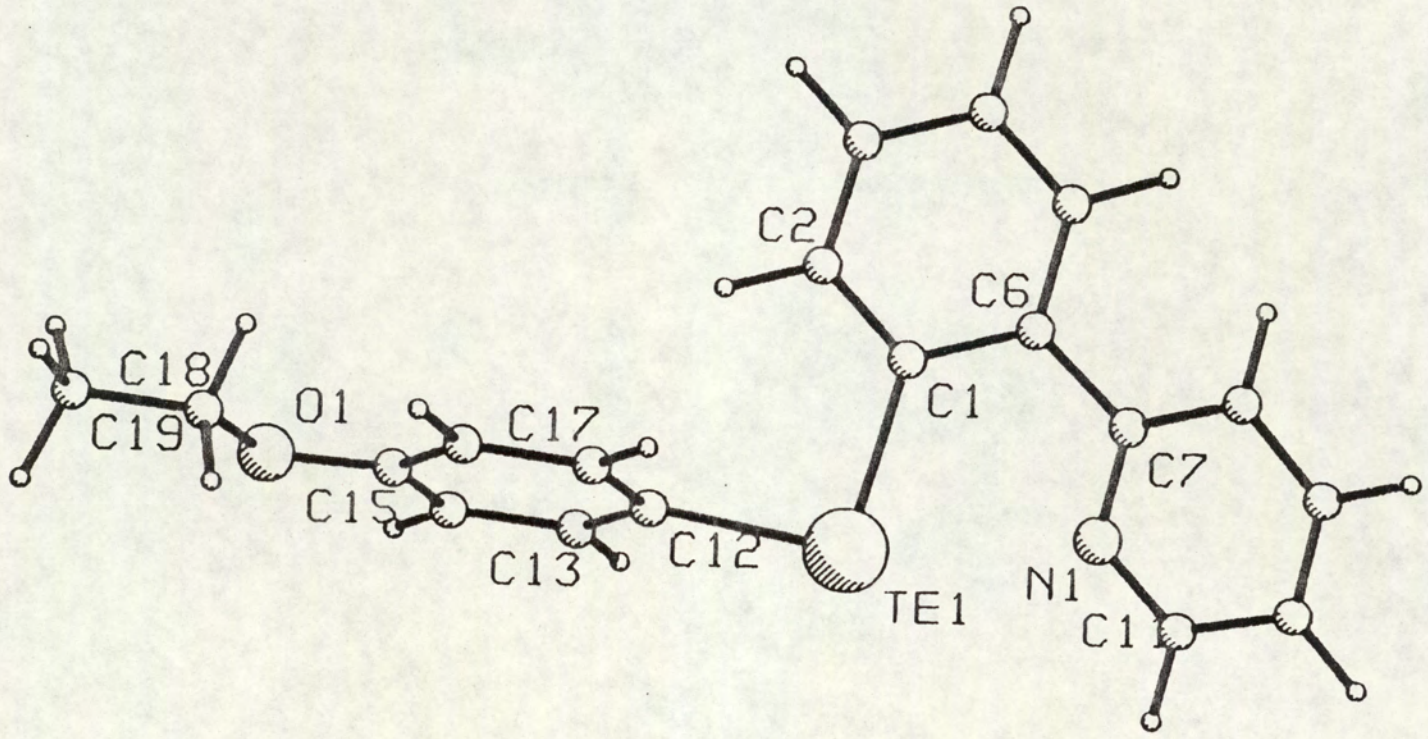
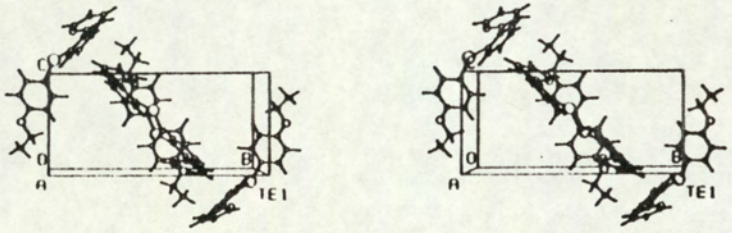


Figure 3.18. Stereoscopic packing diagram of p-ethoxyphenyl[2-(2-pyridyl)phenyl]telluride.





# CHAPTER 4

# GENERAL DISCUSSION

## 4.1 General Discussion

The nine new organotellurium structures studies, fall into three main groups, when characterised solely on the basis of the oxidation state of tellurium, that is 1,2 or 4. In each oxidation state, therefore, at least one lone pair of electrons is located in the valence shell. These structures can be categorised further, by considering the number and type of ligands which are attached to the tellurium atom(s).

Using this additional means of classification, the four $Te^{IV}$ structures studied can be sub-divided into three types, $RTeX_3$, $R_2TeX_2$ and $RR^+TeX_2X^-$ (Where R = an aromatic or aliphatic group and X = Br or Cl). In each structure the coordination sphere around tellurium is basically octahedral, with a vacant equatorial site. The ligands which surround the central tellurium atom in each structure adopt the preferred octahedral geometry, as specified by Mangion et al[79]. In this environment, the bonding arrangement around tellurium can be visualised in the following way: Tellurium forms a covalent bond with each of the atoms which occupy the two axial sites in the coordination sphere, and is bonded in the equatorial plane to two atoms by single covalent bonds. A fifth interaction occurs, which is longer than the sum of the single bond covalent radii for the participating atoms, but which is significantly shorter than the sum of their van der Waals radii. The fourth equatorial site, may be considered as essentially vacant or occupied by the lone pair of electrons on

tellurium if they are considered to be stereochemically active.

In discussing the general significance of the relative bond lengths around tellurium, consideration needs to be given to the value assigned to the covalent radius of tellurium. Probably the most widely known and universally quoted value, is that assigned by Pauling [108] of 1.37 Å. This quantity can be seen as being composed from two components, relating to the contributions from the core and valence electrons. Van Vechten and Phillips[167], used this philosophy to predict the covalent radius of tellurium of 1.405 Å when in a tetrahedral environment and 1.541 Å when in an octahedral one. The choice of covalent radii assigned to tellurium has been added to by Sanderson[168], who estimated the "non-polar" covalent radius of tellurium to be 1.360 Å. The variety of tellurium radii has been increased still further by McWhinnie and Monsef-Mirzai[40], who considered the question of why axial bonds were longer than equatorial bonds in a significant number of RTeX and $R_2TeX_2$ structures which were described as $\psi$-trigonal bipyramidal. In these systems, they postulated that tellurium is polarised more than the other atoms, (although this assumption may be a poor one in the case of iodine) as it forms bonds to carbon and halide atoms only. Using literature data for such RTeX and $R_2TeX_2$ structures, the pair simply subtracted the Pauling covalent radius of carbon and the relevant halogen(s) from the appropriate bond length involving tellurium, which left a remarkably consistent remainder for the radius of tellurium. Averaging these remainders provided an

estimate of the axial radius, Te(II) 1.54 Å, Te(IV) 1.53 Å and an equatorial radius, Te(II) 1.34 Å, Te(IV) 1.37 Å, plus the view that in many compounds tellurium is best considered as a prolate ellipsoid. It seems, therefore, that given the valence electrons are in directed orbitals, this approach moves away from the spherical model.

If the criteria applied by McWhinnie and Monsef-Mirzai is applied to these four $Te^{IV}$ structures, the average Te equatorial radius works out to be 1.37 Å and the average axial radius is 1.53 Å. These values are identical to those proposed for $Te^{IV}$ by McWhinnie and Monsef-Mirzai[40] and so in principle the combination of the McWhinnie and Monsef-Mirzai radius for tellurium with the appropriate Pauling radius for the bonding atom, gives the best estimate of the "normal" axial and equatorial bond lengths in these four $Te^{IV}$ structures.

The salient features of each structure are presented in table 4.1. It is not proposed to dwell on each bond length in each structure as they have been discussed in full earlier, suffice to say, that the "normal" bonds around tellurium in each structure fall close to the average lengths of bonds for the two elements involved[40,108].

Table 4.1 Salient features of studied structures.

| Compound | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|
| | $R_2Te_3$ | $R_2Te_2$ | $RTeX_3$ | $RTeX_3$ | $R_2TeX_2$ | $R^+RTe_2X^-$ | RTeX | RTeX | $R_2Te$ |
| Te---$Csp^2$ | 2.130 | 2.136<br>2.141 | 2.110 | 2.121 | 2.121<br>2.125 | 2.134 | 2.111 | 2.124<br>2.135 | 2.136<br>2.144 |
| Te---$Csp^3$ | | | | | 2.119<br>2.124 | 2.154 | | | |
| Te---N | 2.554 | 2.882<br>2.876 | 2.244 | 2.422 | | | 2.354 | 2.385<br>2.365 | 2.695 |
| Te---O | | | | | 2.848<br>2.830 | | | | |
| Te---S | | | | | | | 2.518 | 2.569<br>2.543 | |
| $Te_{(axial)}$--X | | | 2.673<br>2.658 | 2.758<br>2.633 | 2.504 2.504<br>2.503 2.512 | 2.527<br>2.487 | | | |
| $Te_{(equatorial)}$-X | | | 2.589 | 2.632 | | 3.338 | | | |
| Te---Te | 2.776 | 2.746 | | | | | | | |
| Inter-molecular distances *trans* to Te-C | | | 3.596 | 3.896 | 3.740<br>3.775 | | | | |
| Intra-molecular distances *trans* to Te--C | | | | | 2.848<br>2.830 | 3.338 | | 3.230<br>3.222 | 2.695 |

A particular interest of this study, was to examine the Lewis acidity of tellurium in various environments. It is well known that mono-organyltellurium(IV) compounds are generally classified into dimers[133,134] or polymers[87,131-133], due to their high Lewis acidity, however a limited number of monomeric or weakly associated compounds are known when the organic group is bulky[135-140].

In an attempt to avoid such intermolecular interactions in the studied mono-organyltellurium(IV) compounds, an electron donating moiety was designed into the organic ligand. It was hoped and anticipated that this donor group incorporated into the system would satisfy the Lewis acidity of the molecule by intramolecular complex formation as opposed to short-range intermolecular interactions.

In the cases of [2-pyridyl]phenyltellurium(IV) tribromide and 2-N,N-methylbenzylamine-C,N')tellurium(IV) tribromide, each can be described as essentially monomeric structures with a short Te...N secondary interaction[39] [2.244 Å, 2.422 Å respectively]. In each structure, no primary bond lies *trans* to the Te-C bond, so presumably if the lone pair of electrons is stereo-chemically active, this site is occupied by this lone pair. If secondary[39] intermolecular interactions are considered, weak contacts occur between tellurium and a symmetry related bromine, which lies approximately *trans* to the Te-C bond [3.596 Å, 3.896 Å]. Therefore it is clear in these two structures, that incorporating an electron donating atom into the organic ligand

appears to satisfy the Lewis acidity of the system by intramolecular autocomplex formation. With this kind of complex formation only very weakly associated or monomeric compounds seem to result, giving an alternative to bulky organic constituents when monomeric or very weakly associated mono-organyltellurium(IV) compounds are required.

In the case of diorganyltellurium(IV) compounds, weak Lewis acidity is usually observed with the majority of well defined examples, showing weak intermolecular interaction in the solid state[184,87,169-172] 'via' secondary bonding[39]. In an effort to examine whether the Lewis acidity of tellurium would be satisfied, by intra or intermolecular interactions, a donor atom was built into one of the organic ligands.

Including dichloro(butyl)tellurobenzaldehyde in this study, may be considered to be drifting away from one of the main themes of this study , as in this instance, the donating atom was oxygen as opposed to nitrogen. This analysis, never-the-less, revealed that the compound contains one intra and one intermolecular interaction as opposed to two intermolecular interactions, which suggests that tellurium seems to prefer intramolecular interactions as opposed to intermolecular interactions to satisfy its Lewis acidity, when given the choice. The Te...O interaction (2.830 and 2.848Å) lies *trans* to the $Te\text{-}C_{(butyl)}$ bond. The positioning of this interaction, even allowing for the Lewis acidity of the central tellurium, seems to have a detrimental effect ie, the Te...O interaction appears to be

significantly weakened by the *trans* influence of the Te-C bond.

As mentioned previously, the analysis of 2-dichlorobutyltelluro-N-dimethylbenzylammonium chloride, was undertaken initially, as the material was considered to be of the type $R_2TeX_2$, with an electron donating nitrogen atom contained in one of the R groups. The structural elucidation revealed the material to be $[RR^+TeCl_2]Cl^-$, with no intramolecular interaction between nitrogen and tellurium presumably due to the nitrogen's protonation. The material never-the-less, exhibited no strong primary interactions *trans* to either Te-C bond, and like the previous stuctures apparently obeys the polarity rule[173], where the more electronegative ligands occupy the axial positions first.

The three tellurium(II) structures investigated can be divided into two types, $R_2Te$ and RTeX (where R = an aliphatic or aromatic group and X = a dithiocarbamate derivative), using the same means of classification as that specified previously to group the tellurium(IV) structures. In each Te(II) structure, the tellurium atom lies at the intersection of a "T", with a Te-C bond forming the stem of the "T"[40].

As previewed in the introduction, tellurium(II) seems to prefer a square planar coordination[41-57], never-the-less several complexes are known where the central tellurium atom is three coordinate; giving a characteristic "T" shape to the compounds[61-67]. The arrangement of the bonds/interactions around tellurium, in these three structures, corresponds to the latter description, where the five electron pairs, (two of which are non bonding) distribute themselves around tellurium to define

a trigonal bipyramid. The two RTeX systems studied have a noticeable difference in geometry about tellurium when secondary intramolecular sulphur interactions are considered. Unlike dimethyldithiocarbamato[2-(2-pyridyl)phenyl]tellurium (II), where the secondary tellurium sulphur interaction [3.667 Å] is twisted away from the N(1)-C(1)-Te(1)-S(1) plane, the same interaction in dimethyldithiocarbamato[2-(2-quinolinyl)phenyl] tellurium(II) [3.222 and 3.230 Å] is in the plane. This phenomenon tends the geometry about tellurium more toward square planar and away from a trigonal bipyramidal in the latter structure. The reason for this difference may simply be due to differences in the packing constraints within each crystal system. As it is difficult to justify this observation on the grounds of any possible difference in the Lewis acidity of the central tellurium in each instance, or on the difference in the *trans* influence of the respective organic ligands. It has been commented upon previously[174], that in about one third of "T" shaped structures, the fourth position in a square planar arrangement around tellurium is approached by a ligand. Dimethyldithiocarbamato-2-(2-quinolinyl)phenyl]tellurium(II) seems to be one of those instances.

Using the subtraction method of McWhinnie and Monsef-Mirzai, the average axial and equatorial radii for tellurium in the two RTeX structures works out to be 1.50 Å and 1.38 Å respectively. These values may not be the same as those reported by McWhinnie and Monsef-Mirzai, (1.54 Å, 1.34 Å), but

they are still reasonably close to support the view that in many compounds tellurium is best considered as a prolate ellipsoid. In the $R_2Te$ structure, on the other hand, there appears to be no significant difference between the axial tellurium carbon bond length and the equatorial tellurium carbon bond length. In this environment, tellurium appears not to have been polarized, and therefore behaves as a more spherical entity.

Relevant data concerning the Lewis acidity of tellurium in this type of environment is somewhat limited, never-the-less what information there is, highlights that mono-organyl tellurium(II) compounds are stronger Lewis acids than their di-organyl counter parts. In this section the shortest tellurium-nitrogen interactions have been demonstrated by dimethyldithiocarbamato[2-(2-pyridyl)phenyl]tellurium(II), (2.365 Å and 2.385 Å). Indicating that the stronger Lewis acid behaviour is being demonstrated in the RTeX structures by tellurium. As the short tellurium nitrogen distances are presumably a consequence of the strong interaction between tellurium and nitrogen caused by tellurium's increased Lewis acidity. This observation is in line with previous work demonstrating tellurium's increased Lewis acidity in RTeX structures as opposed to that in $R_2Te$ structures. The two RTeX structures studied again have no primary bonds *trans* to a Te-C bond, illustrating once more the *trans* influence of Te-C.

It was tempting to dismiss diorganyltellurides from this study, since the materials show quite strong Lewis base behaviour. However, it would be too sweeping to suggest that no

Lewis acid behaviour has been observed[40].

The study of *p*-ethoxyphenyl[2-(2-pyridyl)phenyl]telluride, revealed an intramolecular interaction between tellurium and nitrogen at a distance of 2.695 Å. This distance is longer than all others observed in this study, apart from that found for the equivalent interaction in bis[2-(N-hydroxy)iminophenyl] ditelluride [2.882 Å, 2.876 Å]. This interaction lies *trans* to a Te-C bond, therefore even allowing for the low Lewis acidity of the tellurium in this environment, there appears to be weakening of this interaction by the *trans* influence of Te-C. This situation is akin to that found for 2-dichloro(butyl)tellurobenzaldehyde, where a secondary intra molecular contact is found *trans* to a Te-C bond when tellurium is acting as relatively a poor Lewis acid.

The final group of structures, have been grouped together under the general heading of polytellurides. This heading simply means that each structure studied contained more than one tellurium atom within each individual molecule. The two structures studied in this section can be classified as $R_2Te_2$ and $R_2Te_3$ systems, where R = an aromatic group.

The environment around the terminal tellurium in each system, can be considered to be "T" shaped[40]. This description may be considered a little tenuous in the case of bis[2-(N-hydroxy)iminophenyl] ditelluride, however where the tellurium Te-N distance at 2.876 and 2.882 Å forms part of the proposed "T" shape. In both structures there seems to be no significant discernable difference between the axial and

equatorial single bond radii for tellurium. This conclusion is based on the same means of analysis as that performed for the preceding two groups of structures. The tellurium atoms appear to be spherical in nature, rather than prolate ellipsoids, which seems to indicate that tellurium atoms remain spherical unless bonded to a strong electronegative atom which induces polarization within tellurium giving it a prolate ellipsoid geometry.

In the polytellurides, the formal oxidation state for Te is 1. In a previous survey of diorganyldichalcogenides, there was found to be no definitive examples of coordination in $R_2E_2$ systems, were E= Se or Te. However it was pointed out that there seemed to be no reason why intramolecular coordination should not be possible, with any diaryldichalcogenide containing a basic *ortho*-substituent[40].

In the diaryldichalcogenide investigated during this study, bis[2-(N-hydroxy)iminophenyl] ditelluride, a basic *ortho* substituent has been incorporated into the structure. This feature gave rise to two secondary [39] Te...N interactions, 2.876 and 2.882 Å, which are significantly less than the sum of the van der Waals radii, but noticeably longer than the sum of the Pauling Te-N covalent single bond radii[108]. Never-the-less, whether these distances can be construed as definitive evidence of coordination on the part of each tellurium atom, is surely debatable, although slightly longer interactions, 2.923(2) and 2.962(2) Å in dimethyl bis-[2-(4-nitrobenzylidenimino)-phenyloxy]tellurane [129] where tellurium is in an oxidation state

of 4 were considered to be Te...N coordination bonds. The equatorial positons *trans* to Te-C are occupied by the tellurium's two lone pairs of electrons, so no primary bonds are thus positioned *trans* to Te-C in the diaryldítelluride.

Closely related to diaryldichalcogenides are the diaryl trichalcogenides, $R_2E_3$ where E= Se or Te. In this sector, the study of bis[2-(2-pyridyl)phenyl]tritelluride has been performed. In this structure, the pyridyl ring is positioned *ortho* to the tellurium phenyl ring carbon bond, giving rise to a secondary Te...N interaction of 2.554 Å, between the terminal tellurium and the pyridyl nitrogen. This distance is clearly shorter than the corresponding contact in the previously mentioned diaryl ditelluride. Therefore, unlike bis[2-(N-hydroxy)iminophenyl] ditelluride, the terminally positioned telluriums in the tritelluride seem to be demonstrating some Lewis acid behaviour. This interaction lies approximately half way between that found for mono-organyltellurium(II) species and the afore-mentioned diaryldítelluride, suggesting a tendency towards an oxidation state of +2 for the termimal telluriums. If this is the case, then this would infer the structure of the complex to be that of a bis tellurenyl telluride, where the oxidation state of the telluriums along the chain would be +2, -2, +2. Obviously, this conclusion can only be a tentative one on our part at present, as the evidence it is based on is limited. This tentative conclusion may be borne out by the $Te^{125}$ Mossbauer analysis, however this analysis has not yet been completed. If this conclusion indeed turns out to be

correct, it in turn raises another question, of why is there this difference in Lewis acid behaviour between tellurium in the terminal position in the tritelluride and tellurium in a ditelluride ? There seems at present, no clear reason for this difference and so this question has been left unanswered until more information is available concerning diaryldichalcogenides and diaryltrichalcogenides with an *ortho* electron donating substituent.

The recognition that ligand atoms will distribute themselves on a coordination sphere is consistent with VSEPR theory, but frequent lack of any geometric indication of the presence of electron pairs in the coordination sphere also seems to be well documented. When will an electron pair be inert and when will it not be? The position of the lone pair or pairs of electrons in these structures bears some resemblance to that of the hydrogen atom in earlier crystal structure studies, namely that it is inferred from other structural features rather than observed directly.

From the study of these nine structures, the order of Lewis acidity of tellurium agrees with that specified previously[40] i e

$RTeX_3 > R_2TeX_2$

$RTeX > R_2Te$

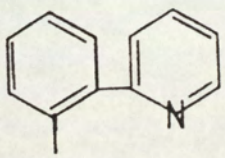
$R_2Te_3 > R_2Te_2$

There is an important and well known danger in the uncritical use of bond distances obtained from X-ray data. The effect of thermal motion of the atoms in a crystal is capable of

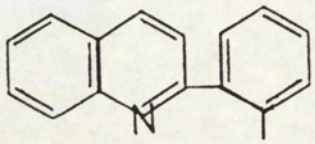
misleading an investigator into thinking that small apparent differences in bond distances are significant. Bearing this fact in mind, the differences between the various tellurium-nitrogen distances can be still considered to be a reflection of the order of Lewis acidity within the structures. That is when tellurium is placed in an environment where it has a high Lewis acidity the tellurium nitrogen distance is relatively short, but when tellurium is in a situation where it has a low Lewis acidity the tellurium nitrogen distance is relatively long. This can be clearly seen by referring to table 4.2, where the tellurium nitrogen interactions are given for each crystal structure studied. In the past, tellurium nitrogen interactions have been considered to be unusual. I would suggest that there is nothing unusual about these tellurium nitrogen interactions.

The major feature of this study is the Lewis acid behaviour of tellurium in different environments, which is reflected by the secondary bonding [39] between tellurium and an electron donating atom, usually nitrogen. This distance also appears to be influenced by the ligand *trans* to it, noticeably in this study, the phenyl group seems to have a strong *trans* influence. Originally the term "*trans* effect" in metal complexes described the influence of a coordinated group upon the practical ease of preparing compounds in which the ligand opposite or *trans* to it had been replaced[175]. Basolo and Pearson defined the *trans* effect as the effect of a coordinated group upon the rate of substitution reactions of ligands opposite to it [175]. Chalt et al proposed the following definition[176]: "the *trans* effect or *trans*

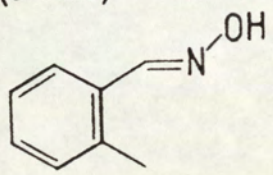
## Table 4.2 Tellurium - Nitrogen Bond Lengths

R = 

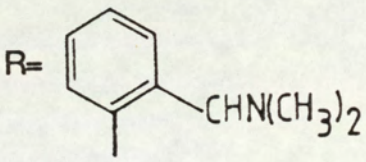
| | Å |
|---|---|
| Te(IV) | |
| $RTeBr_3$ | 2.244 |
| Te(II) | |
| RTe(dmdtc) | 2.354 |
| $RTe(C_6H_4OEt)$ | 2.695 |
| $R_2Te_3$ | 2.554 |

R = 

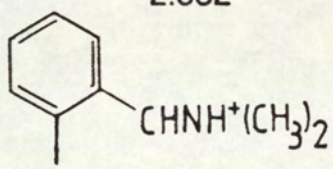
| | Å |
|---|---|
| Te(II) | |
| RTe(dmdtc) | 2.365 |

R= 

| | Å |
|---|---|
| Te(I) | |
| $R_2Te_2$ | 2.876 |
| | 2.882 |

R= 

| | Å |
|---|---|
| Te(IV) | |
| $RTeBr_3$ | 2.422 |

R= 

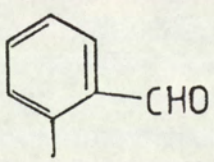
| | Å |
|---|---|
| Te(IV) | |
| $(RR+TeCl_2)Cl^-$ | 4.380 |

R= $C_4H_9$

[N.B. van der Waals distance = 3.61Å sum of Pauling covalent radii = 2.07Å but for "axial" N, figure could be 2.24Å for Te(II) and 2.23Å for Te(IV)]

### Tellurium - Oxygen Interaction

| | Å |
|---|---|
| Te(IV) | |
| $RR'TeCl_2$ | 2.848 |

R= 

R= $CH_2CH_2CH_2CH_3$

[N.B. van der Waals distance = 3.58Å sum of the Pauling covalent radii = 2.03Å]

influence of a group coordinated to a metal ion is the tendency of that group to direct an incoming group into the *trans* position to itself". Quagliano and Schubert used the definition[177]: "the *trans* effect stipulates that the bond holding a group *trans* to an electronegative or otherwise labilizing group is weakened".

This latter description by Quagliano and Schubert, is the most meaningful definition of the *trans* effect with regard to this study. This phenomenon has been widely reported in transition metal chemistry, but these bond length effects are not usually large[178-182]. In a previous review of tellurium(II) complexes where the linear three-centre systems were not symmetrical, pronounced relative *trans* bond lengthening effects of ligands were observed. In addition, it was noted that the phenyl group had a particularly large *trans* bond-lengthening effect[41].

In all of the structures studied in this work, no primary bonds are found *trans* to a Te-C covalent bond. This observation coincides with the earlier observation by O. Vikane[64] who noted, with reference to organotellurium(II) bromide complexes, that only long weak interactions are found *trans* to Te-C bonds. Examples of the *trans* bond lengthening effect of covalently bound carbon manifests itself in organo-tellurium(IV) chlorides, bromides and iodides [81,86,87,103,135,141,149,183].

The two forces of *trans* effect and Lewis acidity may be mutually independent forces, but when the Lewis acidity of tellurium is considered, the *trans* influence of Te-C may also need to be borne in mind. These two factors may be linked by

serendipity, that is, the length of the tellurium nitrogen bond appears dependent on the Lewis acidity of tellurium, this in turn is influenced by the number and type of ligands which are attached to tellurium. Thus, systems with a large number of Te-C bonds, which have a strong reported *trans* effect, possess a tellurium which has a low Lewis acidity and a greater chance of having a Te-C bond *trans* to a secondary interaction.

The Lewis acidity of tellurium in the studied complexes, appears to be satisfied by autocomplex formation, by forming short range intramolecular secondary bonds, with long range weak intermolecular contacts forming in the majority of the $Te^{IV}$ complexes studied.

## 4.2 FUTURE WORK

The main aim of this study was to examine the effects of the tellurium oxidation state and ligand type on the Lewis acidity of tellurium. It became clear during the course of this study that these environmental changes were reflected by the strength of the secondary interactions involving tellurium. Such interactions, usually between tellurium and nitrogen in the majority of structures studies here, appeared to be weakened when a tellurium-carbon bond lay *trans* to the interaction. An interesting area for future study would be to try to remove the supposed *trans* influence of a tellurium-carbon bond on such interactions, by directly bonding the tellurium to the organic ligand via a tellurium-non-carbon bond. A ligand which may prove a possible prospect for such a study is bipyridyl, as it has already been successfully incorporated into a tin(IV) complex[184].

Another area of study is simply to continue this type of study in an effort to enhance the knowledge relating to secondary intramolecular interactions involving tellurium. It is not so long ago after all that Titus et al [152] reported the structure of triphenyltellurium cyanate-chloroform(1/2), and stated "to the best of their knowledge, $Ph_3Te(NCO).^1/_2CHCl_3$ and $Ph_3Te(NCS)$ provided the first structural examples involving secondary tellurium(IV)-nitrogen interactions. The data in this area could be added to by simply expanding such structural studies to include other electron donating groups or atoms such as oxygen or by adding an electron withdrawing group or atoms, such as

fluorine to the organic ligand. The latter modification to the organic ligand or ligands incorportated within the structure, may reduce the *trans* influence of the tellurium-carbon bond. Regrettably, however, such a modification may also increase the Lewis acidity of tellurium, therefore any shortening of the tellurium secondary interactions within such a system may be due to both factors. In particular in this area, it would be interesting to try to incorporate the 2-(2-pyridyl)phenyl ligand into a ditelluride and a diorganyltellurium(IV) dihalide. These two studies would provide a more complete understanding of the effects of tellurium oxidation state and ligand type on the tellurium-nitrogen interaction, centred around just one ligand.

A third possible area of future investigation would be to examine the stability of tritelluride chain complexes, with the aim of increasing the telluride chain length. This length has already been increased to four, as reported by A W Cordes et al [185] and therefore the proliferation of such studies in the future will naturally be constrained by the feasibility of synthesising and crystallising the desired compounds. These constraints may also apply to all these suggestions for future work, and therefore may constrict or prevent their implementation.

# APPENDICES

# APPENDIX A

# UNRESOLVED STRUCTURES

## A1.1 *para* -Ethoxyphenyl-(2-phenylazophenyl-C,N') ditelluride

It was anticipated that the reaction between dimethyldithio -carbamato-(2-phenylazopheyl-C,N')tellurium(II) and the sodium salt of *para*-ethoxyphenyltelluride would yield the first unsymmetrical ditelluride, *para*-ethoxyphenyl -(2-phenylazophenyl) ditelluride. The supposed reaction product appeared unstable when recrystallisation of the material was attempted, in view of this a sample of the raw product was supplied by K Y Abid of this department for analysis. This sample contained both long orange needle-like crystals as well as small cubic deep blood red crystals. On examination the larger of the two types of crystals failed to diffract the X-ray beam when placed on the diffractometer. This fact necessitated that the study of the sample be confined to the smaller red crystals only. A number of the small red crystals were chosen for analysis on the diffractometer, each of which gave essentially the same unit cell. This outcome provided enough confidence to continue with the data collection. Once this was completed, the analysis of the data was commenced on the Birmingham University Multics System. This revealed the crystals to be one of the starting materials, dimethyldithiocarbamato(2-phenylazophenyl-C,N') tellurium(II), which had previously been analysed by M. A. K. Ahmed et al [104]. This result lead to the termination of the analysis at this stage, with the conclusion that the sample supplied contained at "best" mainly unreacted starting materials

as the longer orange crystals were presumed to be the other starting reagent.

**Crystal Data**

This study, a = 6.191(4) Å, b = 12.101(7) Å, c = 21.752(15) Å, ß = 92.52(6)°. Systematic absences consistent with the centrosymmetric space group $P2_1/n$. Position of the tellurium atom, 0.2359(7), 0.1854(4), 0.0976(2).

Ahmed Analysis[104]: a = 6.225(5) Å, b = 12.133(9) Å, c = 21.784(14) Å, ß = 92.88(5)°. Systematic absences consistent with the centrosymmetric space group $P2_1/n$. Position of the tellurium atom 0.2356(1), 0.1868(0), 0.0976(0).

## A1.2 Mercury(II) Complex of a Bis-telluride ligand

## ($C_{20}H_{26}O_3Te_2HgCl_2$)

Very small white crystals were grown from a saturated solution of the product in nitromethane, by N Al-Salim of this department. The crystals although difficult to mount due to their small size (0.275 x 0.075 x 0.050mm) diffracted the X-ray beam on the diffractometer to reveal a C centred monoclinic unit cell (a = 11.860(14) Å, b = 10.656(3) Å, c = 12.188(2)Å, ß = 106.57(4)°). To date, this structure has proved impossible to unravel using the 631 reflection scanned in the range $2°<\theta<25°$, with $I>2\theta(I)$.

This difficulty prompted an analysis of the product by the following alternative physical and analytical techniques: Infrared, Mass Spectrometry, Elemental Analysis, Density and Melting Point. Initially from the results of these studies, it was concluded that the complex had dissociated during recrystallisation with the bis-telluride ligand remaining in the nitromethane and the mercuric chloride precipitating out. This conclusion is somewhat invalidated by the high melting point of the material (above 300°C), therefore an alternative conclusion is that not only the mercury(II) complex decomposes, but so does the bis-telluride ligand. The resulting crystalline material probably then being an oxide of tellurium.

The main aim of this study was to compare the anticipated Te-Hg bonds in this complex with those reported by N Al-Salim et al, for a different bis-telluride mercury(II) complex[106]. In an

attempt to fulfill this original objective, alternative recrystallising solvents were sought. From these, only 2-ethoxyethanol proved to be a suitable solvent, but even this only succeeded in purifying the product. An amorphous white material was deposited from a saturated solution of the complex in this solvent which obviously was unsuitable for X-ray analysis. [Elemental analysis of product deposited from 2-ethoxyethanol; theoretical %age C 28.6, H 3.1, found C 28.2, H 2.8].

## A1.3 Thallium (I) organotellurium (II) Complex

## ($C_{13}H_9O_2TeTl$)

A sample of white crystals grown from a saturated solution of dimethylsulphoxide was supplied for analysis by N Al-Salim of this department. Using a polarising microscope it was clear that the crystals fell into two distinct groups in terms of extinction angles. That is to say, some crystals extinguished at 0° and 90°, whilst others extinguished 45° further on. Ensuring the selection of one of each type of crystal, with regard to extinction angles, the crystallographic analysis was commenced.

Preliminary oscillation photgraphs were taken without any difficulty, but when each type of crystal was placed on the diffractometer, they failed to produce a self consistent unit cell. A number of apparently suitable crystals of each type were tried, but these all failed to Index satisfactorily. The failure of the crystals in this manner had not been foreseen, but was attributed later to either minute surface flaws in the crystals or crystal twinning. This conclusion to the analysis was obviously not a satisfactory one, but all other attempts to recrystallise the material during the course of this work proved fruitless.

No explanation for the two distinct groups of extinguishing angles can be offered and it remains an interesting and somewhat puzzling feature of this study. The "best" primitive cell parameters for the crystal were a = 10.408(36)Å, b = 19.325(20)Å, c = 22.324(73)Å, $\alpha$ = 94.88(33)°, ß = 91.17(52), $\gamma$ = 100.09(14)°, volume 4401.8 $Å^3$ based on seven reflections. In hindsight, it may have been interesting to set a data set running

using this primitive cell, but this option was not taken up at the time.

## A1.4 Bis(4-Hydroxy-3-methylphenyl)tellurium(IV) dichloride, γ-isomer $(C_7H_7O)_2TeCl_2$

A number of samples of tellurium containing complexes were received from the New Delhi Institute of Science and Technology. Amongst these samples, was a glass file which was reported to contain crystals of the γ-isomer of bis(4-hydroxy-3-methylphenyl)tellurium(IV) dichloride. The analysis of this compound was of direct interest in terms of this study with regard to the general themes of secondary bonding and Lewis acidity in tellurium containing systems.

After microscopic analysis of the deep purple crystals, a needle-like crystal was chosen for the X-ray analysis on the Enraf-Nonius CAD-4 diffractometer, using monchromated MoKα radiation. The selected crystal diffracted well when placed on the Birmingham University Weissenberg camera when irradiated with $CuK_\alpha$ radiation.

The initial twenty-five strong reflections scanned in a theta range of 2 - 25° produced an end centred monoclinic cell (a = 14.682(8)Å, b = 12.771(7) Å, c = 8277(5)Å, ß = 92.33(3)° which was remarkably similar to that of the ß isomer previously reported by R H Jones and T A Hamor. (a = 14.657(3) Å, b = 12.787(3) Å, c = 8.279(2) Å, ß = 92.64(2)°)[154]. After careful consideration, it was decided that infact these crystals were not after all the γ-isomer but the ß-isomer. The analysis of the data was terminated at this juncture.

The crystallographic data has been stored on the Birmingham University Multics Computer System, to allow a more in depth analysis of the data should the need arise in the future.

## A1.5 The Perchlorate Salt of bis[2-(2-pyridyl)phenyl] telluride

The analysis of the title salt proceeded smoothly, until the computer analysis stage of the study. At this point, using the cell dimsensions and intensity data collected in conjunction with the assigned space group, failed to yield a self consistent structure. This unexpected set back prompted a more in depth analysis of the crystals by photographic techniques.

This analysis confirmed the dimensions of the unit cell, and the assigned systematic absences for the structure, but failed to highlight or solve the problem encountered in trying to resolve the structure via computer analysis. This difficulty has to date not been overcome and the structure still remains unsolved. The problem in resolving this structure may be due either to considerable disorder within the crystal or to pseudo-symmetry within the unit cell, whatever the course interest in this study has now wained.

In hindsight, the choice of using the perchlorate salt of bis[2-(2-pyridyl)phenyl]telluride was perhaps a poor one, as many perchlorate salts tend to be highly disordered. The analysis of bis[2-(2-pyridyl)phenyl]telluride would have been of great interest, not only in terms of the general themes of study in this work, but also as a direct comparison with bis[2-(2-pyridyl)phenyl]tritelluride. Regrettably, bis[2-(2-pyridyl)phenyl]telluride failed to crystallise in a

variety of crystallising media, whereas its perchlorate salt readily crystallised. This feature, unfortunate as it has turned out, determined the choice of structure for analysis in this case.

Elemental Analysis (%age) Theoretical C 41.5 H 2.53 N 4.40.
Found C 41.6 H 2.60 N 4.10.
Crystal data, Orthorhombic, systematic absences consistent with space group cmca.
Camera: a = 16.765 Å, b = 14.800 Å c = 18.850 Å
Diffractometer: a =16.897(7) Å, b =14.65(4) Å c =19.03(8) Å

# APPENDIX B

# THERMAL PARAMETERS OF STUDIED STRUCTURES

Anisotropic temperature factors ($A^2 \times 10^5$ for tellurium and $A^2 \times 10^4$ for all other non-hydrogen atoms ) with e.s.d.'s in parentheses for Bis[2-(2-pyridyl)phenyl]tritelluride.

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Te(1) | 546( 5) | 462( 4) | 446( 5) | 14( 2) | 203( 3) | -22( 2) |
| Te(2) | 645( 5) | 427( 5) | 571( 5) | 0( 0) | 328( 4) | 0( 0) |
| C(2) | 64( 4) | 52( 5) | 50( 4) | 7( 4) | 13( 3) | 11( 4) |
| C(3) | 68( 5) | 62( 5) | 58( 5) | 0( 4) | 14( 4) | 7( 4) |
| C(4) | 65( 5) | 71( 7) | 85( 8) | -8( 5) | 5( 6) | 17( 5) |
| C(5) | 50( 4) | 57( 5) | 79( 6) | -16( 5) | 29( 4) | -6( 4) |
| C(6) | 62( 4) | 37( 4) | 53( 4) | -19( 3) | 28( 3) | -3( 3) |
| C(1) | 61( 4) | 38( 4) | 49( 5) | -9( 3) | 22( 4) | 0( 3) |
| C(8) | 90( 7) | 64( 5) | 92( 8) | -14( 6) | 52( 6) | -27( 5) |
| C(9) | 127(10) | 61( 6) | 90( 9) | -1( 6) | 66( 8) | -25( 6) |
| C(10) | 137(11) | 62( 6) | 66( 7) | -7( 5) | 46( 7) | -21( 7) |
| C(11) | 106( 7) | 52( 5) | 53( 5) | 1( 4) | 31( 5) | -13( 5) |
| N(1) | 68( 4) | 53( 4) | 48( 4) | -2( 3) | 24( 3) | -13( 3) |
| C(7) | 79( 5) | 40( 4) | 52( 4) | -17( 3) | 42( 4) | -14( 3) |

Anisotropic temperature factors ($A^2 \times 10^5$ for tellurium and $A^2 \times 10^4$ for all other non-hydrogen atoms ) with e.s.d.'s in parentheses for Bis-[2-(N-hydroxy)iminophenyl] Ditelluride

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Te(1) | 475( 4) | 569( 4) | 583( 4) | 17( 2) | 154( 3) | 92( 2) |
| Te(2) | 437( 4) | 562( 4) | 614( 4) | 77( 2) | 142( 3) | 4( 2) |
| O(1) | 111( 6) | 53( 4) | 118( 6) | 3( 4) | 22( 5) | 21( 4) |
| O(2) | 120( 7) | 83( 5) | 130( 7) | 24( 5) | 34( 6) | 15( 5) |
| N(1) | 83( 5) | 51( 4) | 52( 4) | 3( 3) | 3( 3) | 9( 3) |
| N(2) | 75( 5) | 64( 4) | 65( 4) | 12( 3) | 9( 4) | 19( 4) |
| C(1) | 43( 4) | 51( 4) | 48( 4) | 2( 3) | 2( 3) | -3( 3) |
| C(2) | 52( 4) | 57( 4) | 59( 4) | 5( 3) | 12( 3) | 4( 3) |
| C(3) | 46( 4) | 90( 6) | 67( 5) | -9( 5) | 14( 4) | 7( 4) |
| C(4) | 57( 5) | 72( 5) | 56( 5) | -4( 4) | 17( 4) | -11( 4) |
| C(5) | 67( 5) | 66( 5) | 59( 5) | 12( 4) | 18( 4) | -13( 4) |
| C(6) | 44( 4) | 58( 4) | 61( 4) | 6( 3) | -1( 3) | -15( 3) |
| C(7) | 78( 6) | 44( 4) | 70( 5) | -3( 4) | 13( 4) | -1( 4) |
| C(8) | 46( 4) | 56( 4) | 48( 4) | -4( 3) | 13( 3) | 9( 3) |
| C(9) | 50( 5) | 57( 5) | 75( 5) | -1( 4) | 10( 4) | -5( 3) |
| C(10) | 53( 5) | 97( 7) | 68( 5) | -18( 5) | 17( 4) | -19( 5) |
| C(11) | 49( 5) | 100( 8) | 82( 6) | -23( 6) | 11( 4) | -11( 5) |
| C(12) | 53( 5) | 102( 8) | 84( 7) | 1( 6) | 1( 4) | 9( 5) |
| C(13) | 51( 4) | 73( 5) | 56( 5) | -6( 4) | 8( 4) | 10( 4) |
| C(14) | 60( 6) | 74( 6) | 74( 6) | 11( 4) | 0( 4) | 10( 4) |

Anisotropic temperature factors ($A^2 \times 10^4$ for all non-hyrogen atoms) with e.s.d.'s in parentheses for 2-(2-pyridyl)phenyltellurium(IV) tribromide .

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Te(1) | 24( 1) | 26( 1) | 24( 1) | -6( 1) | -1( 1) | 0( 1) |
| Br(1) | 47( 1) | 47( 1) | 48( 2) | -8( 1) | -12( 1) | 5( 1) |
| Br(2) | 66( 2) | 70( 2) | 42( 2) | -6( 1) | -6( 1) | 13( 1) |
| Br(3) | 67( 2) | 63( 2) | 95( 3) | -21( 2) | -15( 2) | -13( 1) |
| C(2) | 37(12) | 32(10) | 44(15) | -1(10) | -10(11) | 12( 9) |
| C(3) | 43(14) | 77(16) | 25(13) | 13(12) | 13(12) | -16(12) |
| C(4) | 50(14) | 69(15) | 31(14) | -22(12) | -3(12) | 1(12) |
| C(5) | 33(11) | 38(10) | 22(12) | -5( 9) | 8(10) | -6( 9) |
| C(6) | 41(12) | 25( 9) | 34(12) | -12( 9) | -18(10) | 0( 8) |
| C(1) | 33(11) | 42(11) | 24(12) | 5( 9) | -3(10) | 8( 9) |
| C(8) | 50(14) | 15( 9) | 104(22) | -6(11) | -34(15) | -2( 9) |
| C(9) | 50(15) | 10( 9) | 112(24) | 1(12) | -34(15) | 1( 9) |
| C(10) | 86(19) | 33(12) | 82(22) | 28(13) | -2(17) | -45(13) |
| C(11) | 34(12) | 80(15) | 22(13) | -1(11) | 7(10) | -18(11) |
| N(1) | 24( 8) | 27( 8) | 33(10) | -6( 7) | 6( 8) | 2( 6) |
| C(7) | 29(10) | 40(10) | 24(11) | -21( 9) | -7( 9) | 8( 8) |

Anisotropic temperature factors ($A^2 \times 10^4$ for all non-hydrogen atoms) with e.s.d.'s in parentheses for (2-N,N-dimethylbenzylamine-C,N')-tellurium(IV) tribromide .

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Te(1) | 38( 1) | 28( 1) | 32( 1) | -1( 1) | 4( 1) | -1( 1) |
| Br(1) | 40( 1) | 54( 1) | 43( 1) | -3( 1) | -6( 1) | 3( 1) |
| Br(2) | 52( 2) | 61( 1) | 104( 2) | -11( 1) | 28( 1) | -12( 1) |
| Br(3) | 100( 2) | 55( 1) | 32( 1) | 5( 1) | 9( 1) | 5( 1) |
| N(1) | 26( 9) | 43( 7) | 33( 9) | 7( 7) | 0( 7) | -1( 7) |
| C(1) | 9( 9) | 30( 8) | 29(10) | 2( 8) | -3( 7) | 1( 6) |
| C(2) | 33(12) | 43(10) | 27(11) | -8( 8) | -4( 9) | 7( 8) |
| C(3) | 44(12) | 34( 9) | 55(13) | -10( 9) | 5(10) | 1( 8) |
| C(4) | 63(16) | 33(10) | 60(16) | -3(10) | 1(13) | 16( 9) |
| C(5) | 62(15) | 36( 9) | 64(14) | 15(11) | 1(11) | 7(10) |
| C(6) | 25(12) | 34( 9) | 52(14) | -2( 8) | -11(10) | -3( 7) |
| C(7) | 46(13) | 36( 8) | 33(12) | 1( 8) | -3(10) | 5( 8) |
| C(8) | 38(15) | 61(12) | 75(16) | -4(12) | -50(12) | -1(10) |
| C(9) | 112(22) | 54(11) | 17(11) | 3( 9) | -12(13) | -2(12) |

Anisotropic temperature factors ($A^2 \times 10^5$ for tellurium and $A^2 \times 10^4$ for all other non-hydrogen atoms ) with e.s.d.'s in parentheses for 2-dichloro(butyl)tellurobenzaldehyde.

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Te(1A) | 471( 4) | 442( 5) | 986( 6) | 88( 4) | -53( 4) | 51( 4) |
| Cl(1A) | 71( 2) | 67( 2) | 110( 3) | -11( 2) | 17( 2) | 8( 2) |
| Cl(2A) | 70( 2) | 145( 4) | 118( 3) | 56( 3) | 17( 2) | 23( 3) |
| O(1A) | 108( 9) | 60( 7) | 178(12) | 2( 8) | -43( 8) | 2( 6) |
| C(1A) | 44( 6) | 32( 6) | 89( 8) | 0( 5) | 8( 6) | 3( 4) |
| C(2A) | 58( 8) | 73(10) | 101( 9) | 27( 8) | 3( 7) | 14( 7) |
| C(3A) | 74( 9) | 53( 8) | 104(10) | 25( 7) | 19( 8) | 20( 7) |
| C(4A) | 64( 8) | 87(11) | 95( 9) | 41( 8) | 12( 7) | 29( 8) |
| C(5A) | 59( 8) | 69( 9) | 90( 8) | 1( 7) | -6( 7) | 6( 7) |
| C(6A) | 53( 7) | 67( 8) | 79( 7) | 6( 6) | -6( 6) | 3( 6) |
| C(7A) | 73(10) | 82(12) | 122(12) | -17(10) | -18( 9) | -1( 8) |
| C(8A) | 59( 8) | 59( 8) | 93( 8) | 2( 7) | -10( 6) | 8( 6) |
| C(9A) | 54( 7) | 82(10) | 103(10) | 6( 8) | -6( 7) | -6( 7) |
| C(10A) | 62( 8) | 86(10) | 64( 7) | -6( 6) | -5( 6) | 1( 7) |
| C(11A) | 81(10) | 113(13) | 106(11) | 35(10) | -15( 8) | -21( 9) |
| Te(1B) | 447( 4) | 351( 4) | 1000( 6) | -50( 4) | -75( 4) | -53( 3) |
| Cl(1B) | 68( 2) | 66( 2) | 109( 2) | 11( 2) | 13( 2) | -11( 2) |
| Cl(2B) | 73( 2) | 105( 3) | 116( 3) | -47( 2) | 14( 2) | -18( 2) |
| O(1B) | 115( 9) | 56( 6) | 164(10) | 11( 7) | -58( 8) | 7( 6) |
| C(1B) | 41( 6) | 50( 7) | 83( 7) | 3( 6) | 7( 5) | -10( 5) |
| C(2B) | 73( 8) | 28( 6) | 85( 7) | -13( 5) | -10( 6) | -5( 5) |
| C(3B) | 76( 9) | 44( 7) | 103( 9) | -15( 7) | 17( 8) | -21( 6) |
| C(4B) | 62( 8) | 59( 8) | 88( 8) | -40( 7) | 8( 7) | -24( 6) |
| C(5B) | 49( 7) | 85(10) | 86( 8) | -13( 8) | 2( 6) | -2( 7) |
| C(6B) | 45( 6) | 48( 7) | 93( 8) | 4( 6) | -3( 6) | 0( 5) |
| C(7B) | 104(12) | 54( 9) | 128(13) | -3( 8) | -49(10) | -4( 8) |
| C(8B) | 60( 7) | 56( 8) | 90( 8) | -16( 6) | -14( 6) | -5( 6) |
| C(9B) | 54( 7) | 39( 6) | 107( 9) | -4( 6) | -8( 7) | 1( 5) |
| C(10B) | 59( 8) | 58( 8) | 83( 8) | 17( 6) | -5( 6) | 4( 6) |
| C(11B) | 69( 9) | 87(11) | 97(10) | -34( 8) | -19( 7) | -4( 8) |

Anisotropic temperature factors ($A^2 \times 10^5$ for tellurium and $A^2 \times 10^4$ for all other non-hydrogen atoms) with e.s.d.'s in parentheses for 2-Dichloro(butyl)telluro-N-dimethylbenzylammonium Chloride

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Te(1) | 275( 2) | 387( 2) | 296( 2) | -6( 2) | 14( 1) | -21( 1) |
| Cl(1) | 47( 1) | 47( 1) | 72( 1) | -12( 1) | -4( 1) | 9( 1) |
| Cl(2) | 69( 1) | 39( 1) | 55( 1) | -6( 1) | 5( 1) | -10( 1) |
| Cl(3) | 44( 1) | 73( 1) | 55( 1) | -20( 1) | -9( 1) | 0( 1) |
| N(1) | 28( 2) | 57( 3) | 32( 2) | 10( 2) | 2( 1) | 4( 2) |
| C(1) | 25( 2) | 33( 2) | 36( 2) | 3( 2) | 1( 2) | -1( 2) |
| C(2) | 40( 2) | 54( 3) | 38( 2) | 8( 3) | 8( 2) | -1( 3) |
| C(3) | 30( 2) | 64( 3) | 65( 3) | 22( 3) | 16( 2) | 2( 3) |
| C(4) | 28( 2) | 59( 3) | 73( 3) | 23( 3) | -4( 2) | -6( 2) |
| C(5) | 38( 2) | 49( 3) | 45( 3) | 12( 3) | -4( 2) | -1( 2) |
| C(6) | 31( 2) | 27( 2) | 37( 2) | 4( 2) | 2( 2) | 0( 2) |
| C(7) | 41( 2) | 45( 3) | 30( 2) | -1( 2) | 2( 2) | 7( 2) |
| C(8) | 52( 3) | 82( 5) | 33( 2) | 12( 3) | 12( 2) | 1( 3) |
| C(9) | 66( 3) | 45( 3) | 63( 3) | -2( 3) | 17( 3) | -9( 3) |
| C(10) | 47( 3) | 50( 3) | 30( 2) | 9( 2) | -2( 2) | -4( 3) |
| C(11) | 65( 3) | 49( 3) | 36( 3) | -1( 2) | 3( 2) | -13( 3) |
| C(12) | 69( 3) | 68( 4) | 38( 3) | 1( 3) | 6( 3) | -14( 3) |
| C(13) | 69( 4) | 91( 5) | 55( 3) | -12( 3) | 18( 3) | -26( 4) |

Anisotropic temperature factors ($A^2 \times 10^5$ for tellurium and $\times 10^4$ for the other non-hydrogen atoms) with e.s.d.'s in parentheses for dimethyldithiocarbamato-2-(2-pyridyl)phenyltellurium(II) .

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Te(1) | 378( 3) | 256( 2) | 453( 3) | 7( 2) | -51( 2) | -141( 2) |
| S(1) | 37( 1) | 40( 1) | 47( 1) | -12( 1) | -3( 1) | -11( 1) |
| S(2) | 47( 1) | 46( 1) | 53( 1) | -18( 1) | 2( 1) | -14( 1) |
| N(1) | 35( 2) | 36( 2) | 42( 2) | -1( 2) | 0( 2) | -12( 2) |
| N(2) | 39( 2) | 54( 3) | 44( 2) | -8( 2) | -6( 2) | -16( 2) |
| C(1) | 35( 2) | 26( 2) | 37( 2) | 0( 2) | 5( 2) | -7( 2) |
| C(2) | 42( 3) | 35( 3) | 44( 3) | 10( 2) | 0( 2) | -9( 2) |
| C(3) | 48( 3) | 33( 3) | 59( 3) | 16( 2) | 1( 3) | -12( 2) |
| C(4) | 60( 3) | 22( 2) | 76( 4) | 6( 2) | 16( 3) | -11( 2) |
| C(5) | 52( 3) | 32( 3) | 58( 3) | -14( 2) | 13( 2) | -18( 2) |
| C(6) | 41( 2) | 25( 2) | 40( 2) | -6( 2) | 8( 2) | -12( 2) |
| C(7) | 38( 2) | 38( 3) | 40( 2) | -8( 2) | 7( 2) | -15( 2) |
| C(8) | 65( 3) | 53( 3) | 49( 3) | -13( 2) | -4( 2) | -27( 3) |
| C(9) | 73( 4) | 77( 4) | 53( 3) | -17( 3) | -14( 3) | -39( 3) |
| C(10) | 58( 3) | 60( 3) | 40( 3) | 5( 2) | -12( 2) | -18( 3) |
| C(11) | 48( 3) | 40( 3) | 45( 3) | 0( 2) | 2( 2) | -12( 2) |
| C(12) | 42( 2) | 27( 2) | 32( 2) | 2( 2) | -1( 2) | -14( 2) |
| C(13) | 43( 3) | 76( 4) | 64( 3) | -4( 3) | -15( 3) | -16( 3) |
| C(14) | 73( 4) | 83( 5) | 57( 3) | -31( 3) | -4( 3) | -38( 4) |

Anisotropic temperature factors ($A^2 \times 10^5$ for tellurium and $A^2 \times 10^4$ for all non-hydrogen atoms) with e.s.d.'s in parentheses for dimethyldithiocarbamato-2-(2-quinolinyl) phenyltellurium(II) .

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Te(1A) | 429( 2) | 407( 1) | 483( 2) | 141( 1) | 51( 1) | 68( 1) |
| S(1A) | 57( 1) | 41( 1) | 53( 1) | 11( 1) | 5( 1) | 13( 1) |
| S(2A) | 74( 1) | 53( 1) | 53( 1) | 9( 1) | -2( 1) | 25( 1) |
| N(1A) | 42( 2) | 41( 2) | 55( 2) | 21( 2) | 6( 1) | 6( 1) |
| N(2A) | 46( 2) | 41( 2) | 68( 2) | 20( 2) | 10( 2) | 9( 1) |
| C(1A) | 38( 2) | 59( 2) | 52( 2) | 22( 2) | 7( 2) | 6( 2) |
| C(2A) | 53( 3) | 60( 3) | 58( 3) | 15( 2) | 9( 2) | 7( 2) |
| C(3A) | 65( 3) | 82( 4) | 53( 3) | 13( 3) | 4( 2) | 9( 3) |
| C(4A) | 65( 3) | 104( 4) | 55( 3) | 29( 3) | -1( 2) | 13( 3) |
| C(5A) | 59( 3) | 89( 4) | 61( 3) | 36( 3) | 4( 2) | 9( 3) |
| C(6A) | 32( 2) | 70( 3) | 61( 3) | 29( 2) | 10( 2) | 10( 2) |
| C(7A) | 33( 2) | 61( 3) | 63( 3) | 33( 2) | 9( 2) | 8( 2) |
| C(8A) | 61( 3) | 72( 3) | 74( 3) | 44( 3) | 15( 2) | 8( 2) |
| C(9A) | 61( 3) | 61( 3) | 92( 4) | 47( 3) | 15( 3) | 5( 2) |
| C(10A) | 36( 2) | 49( 2) | 82( 3) | 29( 2) | 10( 2) | 5( 2) |
| C(11A) | 43( 2) | 42( 2) | 64( 3) | 20( 2) | 7( 2) | 7( 2) |
| C(12A) | 77( 3) | 46( 2) | 59( 3) | 21( 2) | 3( 2) | 8( 2) |
| C(13A) | 92( 4) | 53( 3) | 63( 3) | 12( 2) | 8( 3) | 13( 3) |
| C(14A) | 74( 3) | 49( 3) | 83( 4) | 10( 3) | 10( 3) | 2( 2) |
| C(15A) | 61( 3) | 43( 2) | 101( 4) | 24( 3) | 15( 3) | 2( 2) |
| C(16A) | 34( 2) | 44( 2) | 61( 3) | 19( 2) | 10( 2) | 11( 2) |
| C(17A) | 73( 3) | 75( 3) | 78( 3) | 45( 3) | 14( 3) | 18( 3) |
| C(18A) | 60( 3) | 40( 2) | 81( 3) | 16( 2) | 18( 2) | 10( 2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| Te(1B) | 477( 2) | 423( 2) | 518( 2) | 200( 1) | 58( 1) | 80( 1) |
| S(1B) | 55( 1) | 45( 1) | 62( 1) | 13( 1) | -2( 1) | 11( 1) |
| S(2B) | 67( 1) | 55( 1) | 53( 1) | 17( 1) | 0( 1) | 11( 1) |
| N(1B) | 52( 2) | 53( 2) | 67( 2) | 33( 2) | 18( 2) | 18( 2) |
| N(2B) | 59( 2) | 45( 2) | 60( 2) | 19( 2) | 12( 2) | 13( 2) |
| C(1B) | 47( 2) | 56( 2) | 59( 3) | 37( 2) | 2( 2) | 2( 2) |
| C(2B) | 77( 3) | 65( 3) | 62( 3) | 33( 3) | -3( 3) | 6( 2) |
| C(3B) | 78( 4) | 82( 4) | 76( 4) | 42( 3) | -10( 3) | 7( 3) |
| C(4B) | 83( 4) | 103( 4) | 81( 4) | 62( 4) | -22( 3) | -16( 3) |
| C(5B) | 51( 3) | 101( 4) | 103( 4) | 74( 4) | 10( 3) | 7( 3) |
| C(6B) | 47( 2) | 69( 3) | 77( 3) | 50( 3) | 12( 2) | 9( 2) |
| C(7B) | 50( 3) | 66( 3) | 95( 4) | 57( 3) | 28( 3) | 22( 2) |
| C(8B) | 62( 3) | 77( 4) | 125( 5) | 56( 4) | 40( 3) | 37( 3) |
| C(9B) | 96( 4) | 85( 4) | 144( 6) | 76( 4) | 80( 4) | 61( 4) |
| C(10B) | 92( 4) | 53( 3) | 104( 4) | 48( 3) | 59( 3) | 33( 3) |
| C(11B) | 71( 3) | 48( 2) | 72( 3) | 35( 2) | 33( 2) | 22( 2) |
| C(12B) | 76( 3) | 49( 3) | 71( 3) | 20( 2) | 18( 3) | 19( 2) |
| C(13B) | 118( 5) | 48( 3) | 70( 3) | 21( 3) | 21( 3) | 15( 3) |
| C(14B) | 140( 6) | 49( 3) | 71( 4) | 21( 3) | 36( 4) | 10( 3) |
| C(15B) | 121( 5) | 51( 3) | 104( 5) | 39( 3) | 71( 4) | 36( 3) |
| C(16B) | 48( 2) | 48( 2) | 57( 3) | 27( 2) | 7( 2) | 5( 2) |
| C(17B) | 59( 3) | 82( 3) | 95( 4) | 48( 3) | 9( 3) | 27( 3) |
| C(18B) | 82( 3) | 46( 2) | 70( 3) | 8( 2) | 16( 3) | 19( 2) |

Anisotropic temperature factors ($A^2 \times 10^5$ for tellurium and $\times 10^4$ for all other non-hydrogen atoms ) with e.s.d.'s in parentheses for p-ethoxyphenyl[2-(2-pyridyl)phenyl]telluride.

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Te | 630( 3) | 499( 3) | 434( 3) | -69( 2) | 38( 2) | 68( 2) |
| O(1) | 85( 3) | 84( 4) | 50( 3) | 2( 3) | -10( 3) | -23( 3) |
| N(1) | 63( 3) | 53( 3) | 50( 3) | -1( 3) | -1( 3) | 9( 3) |
| C(1) | 55( 3) | 49( 3) | 36( 3) | 8( 3) | 10( 3) | 9( 3) |
| C(2) | 57( 4) | 62( 4) | 44( 4) | 5( 3) | 8( 3) | 1( 3) |
| C(3) | 61( 4) | 76( 5) | 51( 4) | 18( 4) | 7( 3) | 11( 4) |
| C(4) | 68( 4) | 73( 5) | 60( 5) | 7( 4) | 21( 4) | 22( 4) |
| C(5) | 74( 5) | 65( 5) | 46( 4) | 1( 3) | 19( 3) | 19( 4) |
| C(6) | 63( 4) | 44( 3) | 38( 3) | 1( 3) | 13( 3) | 3( 3) |
| C(7) | 64( 4) | 58( 4) | 24( 3) | 4( 3) | 7( 3) | 5( 3) |
| C(8) | 79( 5) | 66( 5) | 64( 5) | -22( 4) | 20( 4) | -4( 4) |
| C(9) | 78( 5) | 87( 6) | 67( 5) | -32( 5) | 5( 4) | -12( 5) |
| C(10) | 75( 5) | 100( 7) | 47( 4) | -12( 4) | -5( 4) | -12( 5) |
| C(11) | 70( 5) | 75( 5) | 63( 5) | -2( 4) | 1( 4) | 8( 4) |
| C(12) | 65( 4) | 54( 4) | 40( 3) | -9( 3) | 7( 3) | 0( 3) |
| C(13) | 62( 4) | 68( 4) | 45( 4) | 6( 3) | 0( 3) | 3( 3) |
| C(14) | 74( 4) | 51( 4) | 58( 4) | 3( 3) | -2( 4) | -8( 4) |
| C(15) | 51( 4) | 73( 5) | 49( 4) | 0( 3) | -1( 3) | -10( 3) |
| C(16) | 82( 5) | 66( 5) | 53( 4) | 5( 4) | 3( 4) | -14( 4) |
| C(17) | 90( 5) | 52( 4) | 43( 4) | -3( 3) | -4( 3) | -6( 4) |
| C(18) | 87( 5) | 88( 7) | 56( 5) | -1( 4) | -12( 4) | -8( 5) |
| C(19) | 111( 7) | 108( 8) | 76( 6) | 4( 6) | -14( 5) | -30( 6) |

# APPENDIX C

# HYDROGEN COORDINATES OF STUDIED STRUCTURES

Fractional hydrogen coordinates ($x10^4$) and isotropic temperature factors ($A^2x10^3$) with e.s.d.'s in parentheses for Bis[2-(2-pyridyl)phenyl]-tritelluride.

| | x | y | z | Uiso |
|---|---|---|---|---|
| H(2) | 1921 | 3556 | 3190 | 80(11) |
| H(3) | 3592 | 3598 | 4088 | 80(11) |
| H(4) | 4754 | 5147 | 3660 | 80(11) |
| H(5) | 4244 | 6656 | 2335 | 80(11) |
| H(8) | 3909 | 7794 | 1313 | 80(11) |
| H(9) | 3308 | 9154 | -63 | 80(11) |
| H(10) | 1620 | 8974 | -902 | 80(11) |
| H(11) | 532 | 7433 | -366 | 80(11) |

Fractional hydrogen coordinates ($x10^4$) and isotropic temperature factors ($A^2x10^3$) with e.s.d.'s in parentheses for Bis-[2-(N-hydroxy)iminophenyl] Ditelluride.

| | x | y | z | Uiso |
|---|---|---|---|---|
| H(2) | -1633 | 3928 | 1751 | 72(10) |
| H(3) | -2234 | 4342 | 3060 | 72(10) |
| H(4) | -2086 | 1548 | 4135 | 72(10) |
| H(5) | -1181 | -1482 | 3975 | 72(10) |
| H(7) | -387 | -3719 | 3176 | 123(22) |
| H(9) | -2348 | 182 | 421 | 72(10) |
| H(10) | -3878 | -963 | 39 | 72(10) |
| H(11) | -4909 | 1493 | -839 | 72(10) |
| H(12) | -4367 | 4462 | -1517 | 72(10) |
| H(14) | -3342 | 6995 | -1709 | 123(22) |
| H(15) | 425 | 4335 | 2235 | 183(72) |
| H(16) | -2454 | 9429 | -1977 | 205(76) |

Fractional hydrogen coordinates ($x10^4$) and isotropic temperature factors ($A^2x10^3$) with e.s.d.'s in parentheses for 2-(2-pyridyl)phenyltellurium(IV) tribromide .

| | x | y | z | Uiso |
|---|---|---|---|---|
| H(2) | -5584 | 4462 | 8110 | 58(33) |
| H(3) | -7357 | 5825 | 9476 | 58(33) |
| H(4) | -6254 | 8536 | 9804 | 58(33) |
| H(5) | -2891 | 9285 | 9123 | 58(33) |
| H(8) | -299 | 10296 | 8145 | 187(84) |
| H(9) | 3177 | 10671 | 7234 | 187(84) |
| H(10) | 4296 | 9198 | 5759 | 187(84) |
| H(11) | 2929 | 6487 | 5884 | 187(84) |

Fractional hydrogen coordinates ($x10^4$) and isotropic temperature factors ($A^2x10^3$) with e.s.d.'s in parentheses for (2-N,N-dimethylbenzylamine-C,N')tellurium(IV) tribromide .

| | x | y | z | Uiso |
|---|---|---|---|---|
| H(2) | 1887 | 2227 | 2217 | 58(28) |
| H(3) | 2095 | 644 | 2506 | 58(28) |
| H(4) | 1845 | -17 | 4704 | 58(28) |
| H(5) | 1152 | 838 | 6747 | 58(28) |
| H(7A) | 1353 | 2309 | 7857 | 52(36) |
| H(7B) | -149 | 2753 | 6842 | 52(36) |
| H(8A) | 3775 | 2885 | 6702 | 116(38) |
| H(8B) | 3406 | 3220 | 8388 | 116(38) |
| H(8C) | 3857 | 3993 | 7146 | 116(38) |
| H(9A) | -64 | 4228 | 7189 | 116(38) |
| H(9B) | 1605 | 4788 | 7423 | 116(38) |
| H(9C) | 1154 | 4015 | 8665 | 116(38) |

Fractional atomic coordinates ($x10^4$) and isotropic temperature factors ($A^2x10^3$) with e.s.d.'s in parentheses for 2-Dichloro(butyl)-tellurobenzaldehyde.

| | x | y | z | Uiso |
|---|---|---|---|---|
| H(2A) | 1646 | 5780 | 655 | 77(19) |
| H(3A) | 1031 | 8051 | 29 | 77(19) |
| H(4A) | 190 | 7531 | -1055 | 77(19) |
| H(5A) | -4 | 4714 | -1551 | 77(19) |
| H(7A) | 196 | 1908 | -1516 | 97(44) |
| H(8A1) | 2076 | 4076 | 1742 | 80(16) |
| H(8A2) | 2531 | 4358 | 839 | 80(16) |
| H(9A1) | 3088 | 1838 | 1185 | 80(16) |
| H(9A2) | 2621 | 1488 | 2068 | 80(16) |
| H(10A) | 3429 | 4442 | 1871 | 80(16) |
| H(A10) | 3001 | 3943 | 2776 | 80(16) |
| H(11A) | 4096 | 3184 | 3032 | 136(36) |
| H(A11) | 4051 | 1929 | 2103 | 136(36) |
| H(1A1) | 3623 | 1431 | 3007 | 136(36) |
| H(2B) | 8427 | 10270 | 4390 | 77(22) |
| H(3B) | 9048 | 12419 | 5059 | 77(22) |
| H(4B) | 9844 | 11850 | 6164 | 77(22) |
| H(5B) | 10012 | 9009 | 6668 | 77(22) |
| H(7B) | 9817 | 6172 | 6565 | 97(30) |
| H(8B1) | 7994 | 8518 | 3242 | 80(19) |
| H(8B2) | 7553 | 8854 | 4155 | 80(19) |
| H(9B1) | 6974 | 6360 | 3824 | 80(19) |
| H(9B2) | 7412 | 6041 | 2907 | 80(19) |
| H(10B) | 6606 | 8934 | 3209 | 80(19) |
| H(B10) | 7034 | 8576 | 2286 | 80(19) |
| H(11B) | 5949 | 7865 | 2006 | 136(32) |
| H(B11) | 5992 | 6473 | 2879 | 136(32) |
| H(1B1) | 6420 | 6115 | 1957 | 136(32) |

Fractional atomic coordinates ($x10^4$) and isotropic temperature factors ($A^2x10^3$) with e.s.d.'s in parentheses for 2-Dichloro(butyl)telluro-N-dimethylbenzyl ammonium Chloride

| | x | y | z | Uiso |
|---|---|---|---|---|
| H(1) | 2699 | 3390 | -1642 | 50(15) |
| H(2) | 2244 | 1417 | 2143 | 74( 9) |
| H(3) | 587 | 1378 | 1567 | 74( 9) |
| H(4) | 181 | 1538 | -109 | 74( 9) |
| H(5) | 1416 | 1745 | -1195 | 74( 9) |
| H(7A) | 3961 | 1613 | -529 | 82( 7) |
| H(7B) | 3150 | 708 | -1337 | 82( 7) |
| H(8A) | 4525 | 2887 | -2029 | 105( 8) |
| H(8B) | 3751 | 4205 | -2647 | 105( 8) |
| H(8C) | 3545 | 2123 | -2688 | 105( 8) |
| H(9A) | 4210 | 4468 | -566 | 105( 8) |
| H(9B) | 3036 | 4818 | -311 | 105( 8) |
| H(9C) | 3489 | 5713 | -1292 | 105( 8) |
| H(10A) | 4583 | -30 | 2924 | 82( 7) |
| H(10B) | 3373 | -182 | 2610 | 82( 7) |
| H(11A) | 4337 | 2673 | 3499 | 82( 7) |
| H(11B) | 3123 | 2436 | 3229 | 82( 7) |
| H(12A) | 4323 | 438 | 4552 | 82( 7) |
| H(12B) | 3132 | 40 | 4237 | 82( 7) |
| H(13A) | 3323 | 1411 | 5732 | 105( 8) |
| H(13B) | 2710 | 2606 | 4891 | 105( 8) |
| H(13C) | 3901 | 3004 | 5206 | 105( 8) |

Fractional atomic coordinates ($x10^4$) and isotropic temperature factors ($A^2x10^3$) with e.s.d.'s in parentheses for dimethyldithiocarbamato-2-(2-pyridyl)phenyltellurium(II).

| | x | y | z | Uiso |
|---|---|---|---|---|
| H(13A) | 3475 | 3219 | -52 | 299(59) |
| H(13B) | 3668 | 4824 | 606 | 299(59) |
| H(13C) | 3490 | 3198 | 1593 | 299(59) |
| H(14A) | 5532 | 1567 | -866 | 116(19) |
| H(14B) | 7235 | 197 | 109 | 116(19) |
| H(14C) | 7397 | 1835 | -871 | 116(19) |
| H(2) | 8909 | 4579 | 1277 | 76(21) |
| H(3) | 9113 | 6997 | 893 | 152(42) |
| H(4) | 10444 | 7878 | 2249 | 55(16) |
| H(5) | 11918 | 6118 | 4042 | 72(22) |
| H(8) | 13575 | 4285 | 5539 | 111(29) |
| H(9) | 14807 | 2119 | 7161 | 134(36) |
| H(10) | 14728 | -287 | 7199 | 85(23) |
| H(11) | 13023 | -860 | 5818 | 39(13) |

Fractional atomic coordinates ($x10^4$) and isotropic temperature factors ($A^2 x10^3$) with e.s.d.'s in parentheses for dimethyldithiocarbamato-2-(2-quinolinyl)phenyltellurium(II) .

| | x | y | z | Uiso |
|---|---|---|---|---|
| H(2A) | 4426 | 9132 | 1814 | 84( 5) |
| H(3A) | 5375 | 9271 | 3218 | 84( 5) |
| H(4A) | 5107 | 7802 | 3509 | 84( 5) |
| H(5A) | 4018 | 6168 | 2373 | 84( 5) |
| H(8A) | 2947 | 4841 | 1421 | 84( 5) |
| H(9A) | 1865 | 3328 | 221 | 84( 5) |
| H(12A) | 2513 | 5580 | -1574 | 84( 5) |
| H(13A) | 1440 | 4057 | -2827 | 84( 5) |
| H(14A) | 550 | 2507 | -2717 | 84( 5) |
| H(15A) | 947 | 2425 | -1374 | 84( 5) |
| H(A17) | 1663 | 10972 | -1081 | 196(15) |
| H(217) | 577 | 9740 | -1579 | 196(15) |
| H(317) | 2959 | 10016 | -1586 | 196(15) |
| H(A18) | 2122 | 11612 | 173 | 196(15) |
| H(218) | 3774 | 11149 | 632 | 196(15) |
| H(318) | 1423 | 10913 | 721 | 196(15) |
| H(2B) | 4374 | 8787 | 6892 | 104( 6) |
| H(3B) | 7223 | 9134 | 7830 | 104( 6) |
| H(4B) | 9660 | 8114 | 7376 | 104( 6) |
| H(5B) | 9261 | 6701 | 6011 | 104( 6) |
| H(8B) | 8885 | 5562 | 4837 | 104( 6) |
| H(9B) | 8066 | 4160 | 3531 | 104( 6) |
| H(12B) | 1328 | 5503 | 3501 | 104( 6) |
| H(13B) | 349 | 4074 | 2173 | 104( 6) |
| H(14B) | 2561 | 2894 | 1547 | 104( 6) |
| H(15B) | 5648 | 3152 | 2232 | 104( 6) |
| H(17B) | -3425 | 10287 | 5696 | 155(12) |
| H(172) | -3856 | 8989 | 5182 | 155(12) |
| H(173) | -2620 | 9666 | 4727 | 155(12) |

| | | | | |
|---|---|---|---|---|
| H(18B) | -1561 | 10885 | 6725 | 155(12) |
| H(182) | 761 | 10745 | 6590 | 155(12) |
| H(183) | -504 | 10060 | 7029 | 155(12) |

Fractional atomic coordinates ($x10^4$) and isotropic temperature factors ($A^2x10^3$) with e.s.d.'s in parentheses for p-ethoxyphenyl-[2-(2-pyridyl)phenyl] telluride.

| | x | y | z | Uiso |
|---|---|---|---|---|
| H(2) | 3484 | -510 | -669 | 80( 7) |
| H(3) | 4800 | -1372 | -1875 | 80( 7) |
| H(4) | 4386 | -2337 | -4208 | 80( 7) |
| H(5) | 2694 | -2514 | -5213 | 80( 7) |
| H(8) | 1204 | -2932 | -5134 | 80( 7) |
| H(9) | -527 | -3023 | -6146 | 80( 7) |
| H(10) | -1644 | -1849 | -5820 | 80( 7) |
| H(11) | -984 | -625 | -4382 | 80( 7) |
| H(13) | 2252 | 1404 | -386 | 80( 7) |
| H(14) | 3194 | 2008 | 2041 | 80( 7) |
| H(16) | 3105 | -251 | 4726 | 80( 7) |
| H(17) | 2122 | -851 | 2277 | 80( 7) |
| H(18A) | 3296 | 668 | 7011 | 114(24) |
| H(18B) | 4370 | 292 | 6000 | 114(24) |
| H(19A) | 4795 | 892 | 8508 | 174(29) |
| H(19B) | 4189 | 1824 | 8084 | 174(29) |
| H(19C) | 5266 | 1560 | 6933 | 174(29) |

# APPENDIX D

# STRUCTURE FACTORS OF STUDIED STRUCTURES

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Bis[2-(2-pyridyl)phenyl]tritelluride



| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 4 | 0 | 0 | 590 | 772 | 8 | 4 | 0 | 1593 | -1539 | 6 | 10 | 0 | 355 | -360 | -8 | 3 | 1 | 136 | 150 | 10 | 5 | 1 | 281 | 285 |
| 6 | 0 | 0 | 899 | -1035 | 10 | 4 | 0 | 274 | -260 | 1 | 11 | 0 | 583 | -627 | -6 | 3 | 1 | 438 | -389 | 14 | 5 | 1 | 223 | 214 |
| 8 | 0 | 0 | 640 | -710 | 12 | 4 | 0 | 398 | 412 | -16 | 1 | 1 | 185 | -171 | -4 | 3 | 1 | 168 | -171 | -9 | 6 | 1 | 441 | 406 |
| 10 | 0 | 0 | 200 | 196 | 14 | 4 | 0 | 509 | 525 | -14 | 1 | 1 | 267 | -249 | -2 | 3 | 1 | 1159 | 1080 | -7 | 6 | 1 | 751 | 719 |
| 12 | 0 | 0 | 1140 | 1135 | 1 | 5 | 0 | 3217 | -3109 | -12 | 1 | 1 | 420 | -426 | 0 | 3 | 1 | 819 | 758 | -5 | 6 | 1 | 861 | 843 |
| 14 | 0 | 0 | 1113 | 1028 | 3 | 5 | 0 | 808 | -824 | -10 | 1 | 1 | 517 | -562 | 2 | 3 | 1 | 975 | 872 | -3 | 6 | 1 | 790 | 781 |
| 16 | 0 | 0 | 588 | 500 | 7 | 5 | 0 | 858 | 793 | -8 | 1 | 1 | 909 | -1169 | 4 | 3 | 1 | 495 | 489 | -1 | 6 | 1 | 230 | 227 |
| 1 | 1 | 0 | 3738 | -3525 | 9 | 5 | 0 | 280 | 257 | -6 | 1 | 1 | 886 | -1076 | 6 | 3 | 1 | 160 | 156 | 1 | 6 | 1 | 313 | -309 |
| 3 | 1 | 0 | 1921 | -1863 | 11 | 5 | 0 | 382 | -371 | -4 | 1 | 1 | 1646 | -1694 | 8 | 3 | 1 | 165 | -120 | 3 | 6 | 1 | 244 | 244 |
| 5 | 1 | 0 | 545 | 639 | 13 | 5 | 0 | 787 | -771 | -2 | 1 | 1 | 2631 | -2556 | 14 | 3 | 1 | 165 | 205 | 5 | 6 | 1 | 266 | 250 |
| 7 | 1 | 0 | 1696 | 2046 | 0 | 6 | 0 | 2625 | 2570 | 0 | 1 | 1 | 708 | -669 | -15 | 4 | 1 | 269 | -287 | 7 | 6 | 1 | 706 | 698 |
| 9 | 1 | 0 | 717 | 843 | 2 | 6 | 0 | 1773 | 1777 | 2 | 1 | 1 | 1398 | -1314 | -13 | 4 | 1 | 553 | -583 | 9 | 6 | 1 | 457 | 421 |
| 11 | 1 | 0 | 345 | -406 | 4 | 6 | 0 | 537 | 547 | 4 | 1 | 1 | 1229 | -1302 | -11 | 4 | 1 | 567 | -553 | 11 | 6 | 1 | 484 | 441 |
| 13 | 1 | 0 | 963 | -960 | 6 | 6 | 0 | 659 | -624 | 6 | 1 | 1 | 1326 | -1476 | -9 | 4 | 1 | 615 | -622 | -10 | 7 | 1 | 246 | -231 |
| 15 | 1 | 0 | 768 | -662 | 8 | 6 | 0 | 661 | -634 | 8 | 1 | 1 | 517 | -663 | -7 | 4 | 1 | 454 | -476 | -8 | 7 | 1 | 783 | -766 |
| 0 | 2 | 0 | 2490 | 2183 | 10 | 6 | 0 | 232 | 219 | 10 | 1 | 1 | 550 | -638 | -5 | 4 | 1 | 558 | -546 | -6 | 7 | 1 | 1193 | -1206 |
| 2 | 2 | 0 | 1401 | 1221 | 12 | 6 | 0 | 666 | 628 | 12 | 1 | 1 | 430 | -454 | -3 | 4 | 1 | 1511 | -1390 | -4 | 7 | 1 | 860 | -876 |
| 4 | 2 | 0 | 1364 | -1334 | 14 | 6 | 0 | 621 | 580 | 14 | 1 | 1 | 211 | -203 | -1 | 4 | 1 | 1356 | -1330 | -2 | 7 | 1 | 814 | -824 |
| 6 | 2 | 0 | 3256 | -3298 | 1 | 7 | 0 | 1108 | -1125 | 16 | 1 | 1 | 143 | -119 | 1 | 4 | 1 | 2034 | -1904 | 0 | 7 | 1 | 219 | -225 |
| 8 | 2 | 0 | 2067 | -2212 | 3 | 7 | 0 | 97 | -115 | -15 | 2 | 1 | 183 | 183 | 3 | 4 | 1 | 1763 | -1643 | 2 | 7 | 1 | 120 | -103 |
| 10 | 2 | 0 | 608 | -692 | 5 | 7 | 0 | 690 | 666 | -13 | 2 | 1 | 222 | 209 | 5 | 4 | 1 | 1076 | -1060 | 4 | 7 | 1 | 481 | -477 |
| 12 | 2 | 0 | 221 | 256 | 7 | 7 | 0 | 1070 | 1061 | -9 | 2 | 1 | 1047 | 1159 | 7 | 4 | 1 | 647 | -599 | 6 | 7 | 1 | 709 | -711 |
| 14 | 2 | 0 | 540 | 542 | 9 | 7 | 0 | 489 | 488 | -7 | 2 | 1 | 766 | 796 | 11 | 4 | 1 | 402 | -394 | 8 | 7 | 1 | 779 | -743 |
| 16 | 2 | 0 | 145 | 137 | 0 | 8 | 0 | 406 | 436 | -5 | 2 | 1 | 1124 | 1111 | 13 | 4 | 1 | 305 | -308 | 10 | 7 | 1 | 699 | -673 |
| 1 | 3 | 0 | 1088 | -972 | 2 | 8 | 0 | 151 | 158 | -3 | 2 | 1 | 1548 | 1485 | 15 | 4 | 1 | 260 | -265 | 12 | 7 | 1 | 160 | -147 |
| 3 | 3 | 0 | 563 | 594 | 4 | 8 | 0 | 816 | -821 | -1 | 2 | 1 | 1451 | 1345 | -14 | 5 | 1 | 323 | 321 | -7 | 8 | 1 | 524 | 545 |
| 5 | 3 | 0 | 2501 | 2433 | 6 | 8 | 0 | 1199 | -1176 | 1 | 2 | 1 | 1006 | 948 | -12 | 5 | 1 | 403 | 395 | -5 | 8 | 1 | 500 | 490 |
| 7 | 3 | 0 | 3075 | 3002 | 8 | 8 | 0 | 994 | -976 | 3 | 2 | 1 | 969 | 903 | -10 | 5 | 1 | 329 | 317 | -3 | 8 | 1 | 467 | 487 |
| 9 | 3 | 0 | 1533 | 1554 | 10 | 8 | 0 | 281 | -280 | 5 | 2 | 1 | 905 | 921 | -8 | 5 | 1 | 207 | 202 | -1 | 8 | 1 | 193 | -196 |
| 11 | 3 | 0 | 389 | 407 | 1 | 9 | 0 | 339 | -355 | 7 | 2 | 1 | 753 | 773 | -4 | 5 | 1 | 224 | -186 | 1 | 8 | 1 | 298 | -302 |
| 13 | 3 | 0 | 317 | -336 | 3 | 9 | 0 | 139 | 146 | 9 | 2 | 1 | 799 | 847 | -2 | 5 | 1 | 921 | 851 | 3 | 8 | 1 | 182 | -191 |
| 15 | 3 | 0 | 318 | -334 | 5 | 9 | 0 | 581 | 594 | 11 | 2 | 1 | 409 | 464 | 0 | 5 | 1 | 1127 | 1119 | 7 | 8 | 1 | 411 | 412 |
| 0 | 4 | 0 | 2482 | 2343 | 7 | 9 | 0 | 812 | 826 | 13 | 2 | 1 | 167 | 158 | 2 | 5 | 1 | 1277 | 1254 | 9 | 8 | 1 | 308 | 303 |
| 2 | 4 | 0 | 1874 | 1740 | 9 | 9 | 0 | 482 | 471 | 15 | 2 | 1 | 107 | 115 | 4 | 5 | 1 | 984 | 915 | 11 | 8 | 1 | 271 | 257 |
| 4 | 4 | 0 | 910 | -826 | 0 | 10 | 0 | 774 | 822 | -14 | 3 | 1 | 282 | 304 | 6 | 5 | 1 | 218 | 206 | -2 | 9 | 1 | 337 | 351 |
| 6 | 4 | 0 | 1984 | -1896 | 2 | 10 | 0 | 414 | 421 | -10 | 3 | 1 | 462 | 521 | 8 | 5 | 1 | 282 | -283 | 0 | 9 | 1 | 723 | 770 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2 | 9 | 1 | 798 | 834 | 11 | 1 | 2 | 697 | -751 | -12 | 4 | 2 | 864 | -896 | 6 | 6 | 2 | 717 | -717 | 9 | 9 | 2 | 539 | -514 |
| 4 | 9 | 1 | 495 | 520 | 13 | 1 | 2 | 101 | -82 | -10 | 4 | 2 | 898 | -883 | 8 | 6 | 2 | 347 | 321 | -6 | 10 | 2 | 171 | 194 |
| 8 | 9 | 1 | 111 | -107 | 15 | 1 | 2 | 460 | 422 | -8 | 4 | 2 | 112 | -93 | 10 | 6 | 2 | 382 | 379 | -4 | 10 | 2 | 358 | 388 |
| -3 | 10 | 1 | 159 | -175 | -16 | 2 | 2 | 481 | 404 | -6 | 4 | 2 | 1734 | 1677 | 12 | 6 | 2 | 136 | 105 | -2 | 10 | 2 | 274 | 298 |
| -1 | 10 | 1 | 558 | -605 | -14 | 2 | 2 | 100 | -90 | -4 | 4 | 2 | 2446 | 2283 | -13 | 7 | 2 | 288 | 276 | 0 | 10 | 2 | 497 | -530 |
| 1 | 10 | 1 | 582 | -633 | -12 | 2 | 2 | 517 | -575 | -2 | 4 | 2 | 1820 | 1718 | -11 | 7 | 2 | 610 | 575 | 2 | 10 | 2 | 543 | -566 |
| 3 | 10 | 1 | 579 | -620 | -10 | 2 | 2 | 764 | -880 | 0 | 4 | 2 | 600 | -536 | -9 | 7 | 2 | 383 | 369 | 4 | 10 | 2 | 511 | -538 |
| 5 | 10 | 1 | 322 | -328 | -8 | 2 | 2 | 239 | 240 | 2 | 4 | 2 | 2100 | -1955 | -7 | 7 | 2 | 508 | -500 | 6 | 10 | 2 | 109 | -125 |
| 0 | 11 | 1 | 324 | 373 | -6 | 2 | 2 | 2753 | 2767 | 4 | 4 | 2 | 2133 | -1978 | -5 | 7 | 2 | 996 | -1015 | -16 | 1 | 3 | 325 | 256 |
| -14 | 0 | 2 | 703 | -629 | -4 | 2 | 2 | 4113 | 3988 | 6 | 4 | 2 | 180 | -163 | -3 | 7 | 2 | 1278 | -1267 | -14 | 1 | 3 | 293 | 280 |
| -12 | 0 | 2 | 1615 | -1608 | -2 | 2 | 2 | 4720 | 4313 | 8 | 4 | 2 | 1400 | 1345 | -1 | 7 | 2 | 354 | -332 | -12 | 1 | 3 | 621 | 642 |
| -10 | 0 | 2 | 2180 | -2326 | 0 | 2 | 2 | 2189 | -1835 | 10 | 4 | 2 | 941 | 899 | 1 | 7 | 2 | 995 | 986 | -10 | 1 | 3 | 454 | 516 |
| -8 | 0 | 2 | 1073 | -1241 | 2 | 2 | 2 | 1943 | -1705 | 12 | 4 | 2 | 475 | 452 | 3 | 7 | 2 | 888 | 920 | -8 | 1 | 3 | 565 | 690 |
| -6 | 0 | 2 | 719 | 795 | 4 | 2 | 2 | 1433 | -1303 | 14 | 4 | 2 | 144 | -119 | 5 | 7 | 2 | 566 | 556 | -6 | 1 | 3 | 1348 | 1541 |
| -4 | 0 | 2 | 1149 | 1535 | 6 | 2 | 2 | 269 | 298 | -15 | 5 | 2 | 203 | 201 | 7 | 7 | 2 | 250 | -242 | -4 | 1 | 3 | 2107 | 2155 |
| -2 | 0 | 2 | 529 | 660 | 8 | 2 | 2 | 1576 | 1658 | -13 | 5 | 2 | 697 | 674 | 9 | 7 | 2 | 696 | -664 | -2 | 1 | 3 | 466 | 449 |
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| -3 | 6 | 13 | 497 | 522 | 4 | 2 | 14 | 252 | -237 | -10 | 1 | 15 | 393 | 460 | -6 | 0 | 16 | 885 | 1089 | -9 | 5 | 16 | 354 | -352 |
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| -8 | 7 | 13 | 319 | -300 | -13 | 3 | 14 | 511 | -537 | -6 | 1 | 15 | 573 | 588 | -2 | 0 | 16 | 300 | 301 | -5 | 5 | 16 | 803 | -756 |
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| 0 | 7 | 13 | 357 | -394 | -3 | 3 | 14 | 1288 | -1224 | 2 | 1 | 15 | 322 | 324 | -11 | 1 | 16 | 279 | 269 | -6 | 1 | 17 | 248 | -244 |
| -3 | 8 | 13 | 281 | 288 | -1 | 3 | 14 | 1491 | -1454 | 4 | 1 | 15 | 171 | 164 | -9 | 1 | 16 | 144 | -161 | -4 | 1 | 17 | 307 | -287 |
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| -8 | 0 | 14 | 1509 | -1699 | -10 | 4 | 14 | 531 | -507 | -9 | 2 | 15 | 213 | -226 | -1 | 1 | 16 | 342 | 322 | -11 | 2 | 17 | 268 | 279 |
| -6 | 0 | 14 | 847 | -1025 | -8 | 4 | 14 | 847 | -774 | -7 | 2 | 15 | 293 | -280 | 1 | 1 | 16 | 615 | 586 | -9 | 2 | 17 | 303 | 330 |
| -2 | 0 | 14 | 639 | 662 | -6 | 4 | 14 | 321 | -306 | -5 | 2 | 15 | 353 | -328 | 3 | 1 | 16 | 409 | 399 | -7 | 2 | 17 | 227 | 210 |
| 0 | 0 | 14 | 519 | 521 | -4 | 4 | 14 | 440 | 446 | -3 | 2 | 15 | 355 | -355 | -12 | 2 | 16 | 718 | -789 | -5 | 2 | 17 | 170 | 164 |
| 2 | 0 | 14 | 513 | -511 | -2 | 4 | 14 | 1013 | 975 | -1 | 2 | 15 | 325 | -299 | -10 | 2 | 16 | 448 | -459 | -3 | 2 | 17 | 194 | 172 |
| 4 | 0 | 14 | 734 | -787 | 0 | 4 | 14 | 759 | 729 | 1 | 2 | 15 | 536 | -537 | -8 | 2 | 16 | 135 | 129 | -1 | 2 | 17 | 133 | 168 |
| 6 | 0 | 14 | 582 | -706 | 4 | 4 | 14 | 466 | -452 | -10 | 3 | 15 | 107 | -134 | -6 | 2 | 16 | 679 | 660 | 1 | 2 | 17 | 248 | 254 |
| -13 | 1 | 14 | 141 | -109 | -11 | 5 | 14 | 522 | 492 | -8 | 3 | 15 | 278 | -294 | -4 | 2 | 16 | 447 | 403 | -6 | 3 | 17 | 154 | 183 |
| -11 | 1 | 14 | 582 | 652 | -9 | 5 | 14 | 1150 | 1115 | -6 | 3 | 15 | 367 | -372 | -2 | 2 | 16 | 364 | -340 | -4 | 3 | 17 | 212 | 217 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -5 | 4 | 17 | 475 | -466 | -8 | 0 | 18 | 128 | 143 | 0 | 0 | 18 | 423 | -400 | -1 | 1 | 18 | 454 | 436 | -2 | 2 | 18 | 441 | -435 |
| -3 | 4 | 17 | 358 | -360 | -6 | 0 | 18 | 404 | -518 | -9 | 1 | 18 | 453 | -549 | -8 | 2 | 18 | 449 | 423 | -7 | 3 | 18 | 461 | -436 |
| -1 | 4 | 17 | 311 | -301 | -4 | 0 | 18 | 840 | -960 | -5 | 1 | 18 | 492 | 516 | -6 | 2 | 18 | 117 | 129 | -3 | 3 | 18 | 402 | 367 |
| -10 | 0 | 18 | 349 | 340 | -2 | 0 | 18 | 724 | -722 | -3 | 1 | 18 | 847 | 840 | -4 | 2 | 18 | 375 | -370 | | | | | |

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Bis-[2-(n-hydroxy)iminophenyl] ditelluride PAGE 1

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2 | 0 | 0 | 1082 | 963 | 11 | 2 | 0 | 744 | -732 | 14 | 4 | 0 | 166 | 175 | -7 | 0 | 1 | 420 | -389 | 10 | 1 | 1 | 1083 | -1084 |
| 4 | 0 | 0 | 1614 | -1419 | 12 | 2 | 0 | 546 | -546 | 15 | 4 | 0 | 126 | -134 | -5 | 0 | 1 | 1898 | -1645 | 11 | 1 | 1 | 313 | 308 |
| 6 | 0 | 0 | 1054 | -954 | 13 | 2 | 0 | 142 | -155 | 1 | 5 | 0 | 362 | 351 | -3 | 0 | 1 | 1387 | -1164 | 12 | 1 | 1 | 87 | -100 |
| 8 | 0 | 0 | 56 | -36 | 15 | 2 | 0 | 225 | 228 | 2 | 5 | 0 | 351 | 346 | 3 | 0 | 1 | 1506 | 1530 | 13 | 1 | 1 | 118 | -106 |
| 10 | 0 | 0 | 396 | -366 | 16 | 2 | 0 | 256 | 256 | 3 | 5 | 0 | 340 | -310 | 5 | 0 | 1 | 931 | -892 | 15 | 1 | 1 | 382 | -381 |
| 12 | 0 | 0 | 1244 | -1179 | 17 | 2 | 0 | 271 | -262 | 4 | 5 | 0 | 586 | 541 | 7 | 0 | 1 | 516 | 473 | 16 | 1 | 1 | 284 | -281 |
| 14 | 0 | 0 | 264 | -272 | 1 | 3 | 0 | 1129 | 978 | 5 | 5 | 0 | 594 | -547 | 9 | 0 | 1 | 207 | 188 | -17 | 2 | 1 | 337 | 345 |
| 16 | 0 | 0 | 621 | 605 | 2 | 3 | 0 | 1689 | 1547 | 6 | 5 | 0 | 574 | 535 | 11 | 0 | 1 | 1174 | -1119 | -15 | 2 | 1 | 250 | 249 |
| 18 | 0 | 0 | 371 | 384 | 3 | 3 | 0 | 128 | 122 | 7 | 5 | 0 | 251 | 246 | 13 | 0 | 1 | 1002 | -964 | -12 | 2 | 1 | 455 | 436 |
| 1 | 1 | 0 | 913 | 751 | 4 | 3 | 0 | 1391 | 1259 | 8 | 5 | 0 | 203 | 180 | 17 | 0 | 1 | 379 | 389 | -11 | 2 | 1 | 356 | -337 |
| 2 | 1 | 0 | 1481 | 1263 | 5 | 3 | 0 | 388 | -369 | 9 | 5 | 0 | 358 | 354 | -18 | 1 | 1 | 140 | 138 | -10 | 2 | 1 | 1075 | 1034 |
| 3 | 1 | 0 | 1019 | 1267 | 6 | 3 | 0 | 59 | -68 | 10 | 5 | 0 | 119 | -103 | -17 | 1 | 1 | 71 | -75 | -9 | 2 | 1 | 486 | -474 |
| 4 | 1 | 0 | 361 | 364 | 7 | 3 | 0 | 282 | -266 | 11 | 5 | 0 | 169 | -177 | -15 | 1 | 1 | 394 | 389 | -8 | 2 | 1 | 125 | -91 |
| 5 | 1 | 0 | 805 | 787 | 8 | 3 | 0 | 121 | 103 | 12 | 5 | 0 | 228 | -242 | -14 | 1 | 1 | 461 | 457 | -7 | 2 | 1 | 513 | -503 |
| 6 | 1 | 0 | 623 | -574 | 9 | 3 | 0 | 128 | -145 | 13 | 5 | 0 | 160 | -174 | -13 | 1 | 1 | 381 | 364 | -6 | 2 | 1 | 1462 | -1386 |
| 7 | 1 | 0 | 1488 | -1373 | 10 | 3 | 0 | 464 | 502 | 0 | 6 | 0 | 574 | -564 | -12 | 1 | 1 | 529 | 508 | -5 | 2 | 1 | 876 | -1069 |
| 8 | 1 | 0 | 301 | 258 | 11 | 3 | 0 | 347 | -372 | 1 | 6 | 0 | 229 | 203 | -11 | 1 | 1 | 267 | -263 | -4 | 2 | 1 | 812 | -693 |
| 9 | 1 | 0 | 1629 | -1550 | 12 | 3 | 0 | 134 | -136 | 2 | 6 | 0 | 240 | -224 | -10 | 1 | 1 | 894 | -829 | -3 | 2 | 1 | 136 | -116 |
| 10 | 1 | 0 | 438 | 415 | 13 | 3 | 0 | 129 | -141 | 3 | 6 | 0 | 532 | 508 | -9 | 1 | 1 | 304 | -267 | -2 | 2 | 1 | 674 | 590 |
| 11 | 1 | 0 | 180 | -179 | 14 | 3 | 0 | 637 | -661 | 4 | 6 | 0 | 236 | 225 | -8 | 1 | 1 | 1986 | -1805 | -1 | 2 | 1 | 1153 | 992 |
| 13 | 1 | 0 | 142 | 142 | 15 | 3 | 0 | 165 | 168 | 5 | 6 | 0 | 164 | 156 | -7 | 1 | 1 | 1042 | 921 | 0 | 2 | 1 | 763 | -686 |
| 14 | 1 | 0 | 430 | -414 | 16 | 3 | 0 | 372 | -387 | 6 | 6 | 0 | 82 | 67 | -6 | 1 | 1 | 895 | -784 | 1 | 2 | 1 | 1602 | 1404 |
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| 16 | 1 | 0 | 241 | -241 | 1 | 4 | 0 | 85 | -87 | 8 | 6 | 0 | 178 | -155 | -4 | 1 | 1 | 204 | 169 | 3 | 2 | 1 | 1183 | 1014 |
| 18 | 1 | 0 | 116 | 117 | 2 | 4 | 0 | 415 | 380 | 9 | 6 | 0 | 98 | 91 | -3 | 1 | 1 | 720 | -611 | 4 | 2 | 1 | 619 | 518 |
| 0 | 2 | 0 | 1642 | 1384 | 3 | 4 | 0 | 210 | 167 | 1 | 7 | 0 | 65 | -71 | -2 | 1 | 1 | 118 | 137 | 5 | 2 | 1 | 76 | 60 |
| 1 | 2 | 0 | 865 | -757 | 4 | 4 | 0 | 827 | 741 | 2 | 7 | 0 | 160 | -163 | -1 | 1 | 1 | 1583 | -1396 | 6 | 2 | 1 | 1433 | 1583 |
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| 3 | 2 | 0 | 701 | -622 | 6 | 4 | 0 | 116 | 81 | 4 | 7 | 0 | 166 | 163 | 1 | 1 | 1 | 917 | 779 | 8 | 2 | 1 | 731 | 846 |
| 4 | 2 | 0 | 722 | -610 | 7 | 4 | 0 | 812 | 731 | 5 | 7 | 0 | 53 | -33 | 2 | 1 | 1 | 2268 | 2065 | 9 | 2 | 1 | 374 | -389 |
| 5 | 2 | 0 | 1644 | 1566 | 8 | 4 | 0 | 638 | -591 | 6 | 7 | 0 | 398 | 396 | 3 | 1 | 1 | 2253 | 2316 | 10 | 2 | 1 | 506 | -530 |
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| 8 | 2 | 0 | 408 | -407 | 11 | 4 | 0 | 379 | -425 | -13 | 0 | 1 | 595 | -564 | 7 | 1 | 1 | 414 | -423 | 13 | 2 | 1 | 394 | -415 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 16 | 2 | 1 | 71 | -72 | -14 | 4 | 1 | 78 | 80 | -5 | 5 | 1 | 419 | -391 | 8 | 6 | 1 | 144 | 146 | -13 | 1 | 2 | 952 | 920 |
| 17 | 2 | 1 | 182 | 194 | -13 | 4 | 1 | 398 | 424 | -4 | 5 | 1 | 639 | -585 | 9 | 6 | 1 | 149 | -133 | -12 | 1 | 2 | 223 | 199 |
| -17 | 3 | 1 | 72 | -76 | -12 | 4 | 1 | 404 | 448 | -3 | 5 | 1 | 524 | -490 | 10 | 6 | 1 | 260 | 258 | -11 | 1 | 2 | 432 | 416 |
| -16 | 3 | 1 | 277 | 285 | -11 | 4 | 1 | 159 | 185 | -2 | 5 | 1 | 118 | 107 | -6 | 7 | 1 | 100 | 94 | -10 | 1 | 2 | 303 | -297 |
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| -12 | 3 | 1 | 148 | -156 | -7 | 4 | 1 | 676 | -623 | 2 | 5 | 1 | 84 | 54 | 1 | 7 | 1 | 234 | -229 | -6 | 1 | 2 | 887 | 757 |
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| 0 | 3 | 1 | 1257 | 1081 | 5 | 4 | 1 | 668 | 619 | -11 | 6 | 1 | 145 | 152 | -6 | 0 | 2 | 1875 | -1597 | 6 | 1 | 2 | 423 | -501 |
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| 3 | 3 | 1 | 1199 | 1087 | 8 | 4 | 1 | 761 | 710 | -8 | 6 | 1 | 121 | 117 | 0 | 0 | 2 | 1773 | 1722 | 9 | 1 | 2 | 1092 | -1148 |
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| 7 | 3 | 1 | 156 | -167 | 13 | 4 | 1 | 183 | 182 | -3 | 6 | 1 | 200 | 182 | 8 | 0 | 2 | 1187 | 1247 | 13 | 1 | 2 | 98 | -114 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FÓ | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -12 | 2 | 2 | 66 | -73 | -9 | 3 | 2 | 140 | 128 | -3 | 4 | 2 | 338 | -319 | 8 | 5 | 2 | 345 | 329 | -15 | 0 | 3 | 683 | 635 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 5 | 1 | 15 | 83 | -81 | 4 | 3 | 15 | 59 | -54 | -3 | 1 | 16 | 215 | 191 | 3 | 3 | 16 | 107 | -109 | -3 | 3 | 17 | 178 | 194 |
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| -10 | 2 | 15 | 291 | 283 | -4 | 4 | 15 | 69 | 73 | 5 | 1 | 16 | 165 | 170 | 3 | 0 | 17 | 322 | -324 | -6 | 1 | 18 | 110 | 106 |
| -9 | 2 | 15 | 233 | 235 | -3 | 4 | 15 | 119 | 120 | 6 | 1 | 16 | 133 | -116 | 5 | 0 | 17 | 302 | -247 | -5 | 1 | 18 | 233 | 222 |
| -8 | 2 | 15 | 263 | 256 | -2 | 4 | 15 | 166 | 167 | -10 | 2 | 16 | 120 | 118 | -10 | 1 | 17 | 168 | 179 | -3 | 1 | 18 | 397 | 365 |
| -7 | 2 | 15 | 279 | 267 | -1 | 4 | 15 | 118 | -126 | -9 | 2 | 16 | 195 | 195 | -9 | 1 | 17 | 158 | -154 | 1 | 1 | 18 | 234 | -223 |
| -6 | 2 | 15 | 79 | -85 | 1 | 4 | 15 | 202 | -201 | -8 | 2 | 16 | 341 | 320 | -6 | 1 | 17 | 184 | 173 | 2 | 1 | 18 | 132 | 139 |
| -5 | 2 | 15 | 93 | -102 | 2 | 4 | 15 | 270 | -238 | -6 | 2 | 16 | 255 | 247 | -5 | 1 | 17 | 210 | 202 | -5 | 2 | 18 | 203 | -201 |
| -3 | 2 | 15 | 415 | -414 | -12 | 0 | 16 | 162 | -144 | -5 | 2 | 16 | 101 | -81 | -4 | 1 | 17 | 405 | 368 | -4 | 2 | 18 | 133 | 120 |
| -2 | 2 | 15 | 406 | 404 | -10 | 0 | 16 | 194 | 184 | -3 | 2 | 16 | 285 | 285 | -2 | 1 | 17 | 99 | 89 | -3 | 2 | 18 | 160 | 146 |
| -1 | 2 | 15 | 246 | -231 | -8 | 0 | 16 | 653 | 591 | -2 | 2 | 16 | 217 | -223 | -1 | 1 | 17 | 63 | -81 | -1 | 2 | 18 | 295 | 288 |
| 0 | 2 | 15 | 50 | 45 | | | | | | | | | | | | | | | | | | | | |

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR 2-(2-Pyridyl)phenyltellurium(IV) tribromide 

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 0 | 0 | 894 | 829 | -5 | 3 | 0 | 353 | 368 | 3 | 5 | 0 | 445 | 443 | 2 | -9 | 1 | 175 | 157 | 1 | -5 | 1 | 458 | 393 |
| 2 | 0 | 0 | 41 | -56 | -4 | 3 | 0 | 853 | 906 | 4 | 5 | 0 | 380 | 374 | -4 | -8 | 1 | 95 | -112 | 2 | -5 | 1 | 343 | 302 |
| 3 | 0 | 0 | 458 | 519 | -3 | 3 | 0 | 505 | 281 | 5 | 5 | 0 | 94 | 84 | -3 | -8 | 1 | 148 | 133 | 3 | -5 | 1 | 232 | -193 |
| 4 | 0 | 0 | 142 | -180 | -2 | 3 | 0 | 888 | -910 | 7 | 5 | 0 | 195 | -213 | -2 | -8 | 1 | 101 | 132 | 4 | -5 | 1 | 221 | 218 |
| 5 | 0 | 0 | 365 | -370 | -1 | 3 | 0 | 271 | 296 | -6 | 6 | 0 | 333 | -378 | -1 | -8 | 1 | 252 | 290 | 6 | -5 | 1 | 401 | -387 |
| 6 | 0 | 0 | 156 | -168 | 0 | 3 | 0 | 187 | 224 | -5 | 6 | 0 | 150 | -179 | 0 | -8 | 1 | 260 | 279 | -7 | -4 | 1 | 198 | -242 |
| 8 | 0 | 0 | 264 | 256 | 1 | 3 | 0 | 335 | 56 | -4 | 6 | 0 | 228 | 209 | 1 | -8 | 1 | 81 | 117 | -6 | -4 | 1 | 384 | -426 |
| -6 | 1 | 0 | 302 | 308 | 2 | 3 | 0 | 731 | 742 | -3 | 6 | 0 | 249 | 151 | 2 | -8 | 1 | 183 | -199 | -5 | -4 | 1 | 570 | -584 |
| -5 | 1 | 0 | 1037 | 1033 | 3 | 3 | 0 | 842 | 788 | -2 | 6 | 0 | 649 | 556 | 3 | -8 | 1 | 418 | -435 | -3 | -4 | 1 | 75 | 62 |
| -4 | 1 | 0 | 522 | 486 | 4 | 3 | 0 | 894 | 877 | -1 | 6 | 0 | 782 | 818 | 4 | -8 | 1 | 411 | -386 | -2 | -4 | 1 | 310 | -359 |
| -3 | 1 | 0 | 101 | -119 | 5 | 3 | 0 | 528 | 549 | 0 | 6 | 0 | 373 | 386 | -5 | -7 | 1 | 161 | 211 | -1 | -4 | 1 | 782 | 784 |
| -2 | 1 | 0 | 336 | 378 | 6 | 3 | 0 | 261 | -244 | 1 | 6 | 0 | 181 | 209 | -3 | -7 | 1 | 85 | -97 | 0 | -4 | 1 | 215 | 193 |
| -1 | 1 | 0 | 776 | -776 | 7 | 3 | 0 | 174 | -193 | 2 | 6 | 0 | 272 | -287 | -2 | -7 | 1 | 497 | -484 | 1 | -4 | 1 | 845 | -828 |
| 0 | 1 | 0 | 1514 | -1516 | -7 | 4 | 0 | 163 | -192 | 3 | 6 | 0 | 224 | -258 | -1 | -7 | 1 | 603 | -587 | 2 | -4 | 1 | 673 | -648 |
| 1 | 1 | 0 | 815 | -742 | -6 | 4 | 0 | 71 | -111 | 6 | 6 | 0 | 160 | 169 | 0 | -7 | 1 | 190 | -176 | 3 | -4 | 1 | 634 | -584 |
| 2 | 1 | 0 | 612 | -642 | -5 | 4 | 0 | 91 | -126 | -5 | 7 | 0 | 209 | 219 | 1 | -7 | 1 | 280 | -290 | 4 | -4 | 1 | 232 | -193 |
| 3 | 1 | 0 | 786 | 733 | -4 | 4 | 0 | 450 | -373 | -4 | 7 | 0 | 72 | 74 | 3 | -7 | 1 | 453 | 448 | 5 | -4 | 1 | 98 | 104 |
| 4 | 1 | 0 | 164 | 148 | -3 | 4 | 0 | 302 | 71 | -3 | 7 | 0 | 199 | -186 | 4 | -7 | 1 | 340 | 348 | 6 | -4 | 1 | 95 | 102 |
| 5 | 1 | 0 | 141 | -128 | -2 | 4 | 0 | 611 | 600 | -2 | 7 | 0 | 375 | -379 | 5 | -7 | 1 | 123 | 107 | 7 | -4 | 1 | 384 | 392 |
| 6 | 1 | 0 | 140 | 117 | -1 | 4 | 0 | 679 | 677 | -1 | 7 | 0 | 139 | -158 | -6 | -6 | 1 | 284 | -299 | -6 | -3 | 1 | 440 | 448 |
| 7 | 1 | 0 | 232 | -226 | 0 | 4 | 0 | 506 | 527 | 0 | 7 | 0 | 298 | -318 | -4 | -6 | 1 | 275 | -288 | -5 | -3 | 1 | 228 | 229 |
| 8 | 1 | 0 | 432 | -384 | 1 | 4 | 0 | 377 | -403 | 1 | 7 | 0 | 63 | -16 | -3 | -6 | 1 | 67 | 74 | -4 | -3 | 1 | 490 | -512 |
| -6 | 2 | 0 | 306 | -349 | 2 | 4 | 0 | 317 | -307 | 2 | 7 | 0 | 440 | 506 | -2 | -6 | 1 | 788 | 773 | -3 | -3 | 1 | 229 | -163 |
| -5 | 2 | 0 | 972 | -941 | 3 | 4 | 0 | 535 | -502 | -4 | 8 | 0 | 88 | -91 | -1 | -6 | 1 | 635 | 626 | -2 | -3 | 1 | 376 | -350 |
| -4 | 2 | 0 | 740 | -741 | 4 | 4 | 0 | 835 | -813 | -3 | 8 | 0 | 222 | 215 | 0 | -6 | 1 | 367 | 384 | -1 | -3 | 1 | 539 | -501 |
| -3 | 2 | 0 | 422 | -458 | 5 | 4 | 0 | 186 | -217 | 1 | 8 | 0 | 356 | -360 | 1 | -6 | 1 | 271 | 348 | 0 | -3 | 1 | 189 | 235 |
| -2 | 2 | 0 | 303 | 307 | 6 | 4 | 0 | 159 | 160 | 2 | 8 | 0 | 343 | -406 | 3 | -6 | 1 | 161 | -80 | 1 | -3 | 1 | 419 | 395 |
| -1 | 2 | 0 | 79 | 26 | -6 | 5 | 0 | 148 | 185 | 3 | 8 | 0 | 244 | -293 | 4 | -6 | 1 | 234 | -225 | 2 | -3 | 1 | 1416 | 1387 |
| 0 | 2 | 0 | 377 | -448 | -5 | 5 | 0 | 77 | 94 | 4 | 8 | 0 | 154 | -168 | 5 | -6 | 1 | 117 | -45 | 3 | -3 | 1 | 1399 | 1351 |
| 1 | 2 | 0 | 1006 | 1037 | -4 | 5 | 0 | 251 | -225 | -2 | 9 | 0 | 144 | -148 | 6 | -6 | 1 | 303 | 260 | 4 | -3 | 1 | 373 | 83 |
| 2 | 2 | 0 | 152 | -119 | -3 | 5 | 0 | 206 | -202 | -1 | 9 | 0 | 182 | -144 | -6 | -5 | 1 | 298 | 330 | 5 | -3 | 1 | 232 | 110 |
| 3 | 2 | 0 | 1374 | -1334 | -2 | 5 | 0 | 556 | -558 | 0 | 9 | 0 | 204 | 199 | -5 | -5 | 1 | 308 | 314 | 6 | -3 | 1 | 109 | 71 |
| 4 | 2 | 0 | 263 | -229 | -1 | 5 | 0 | 1340 | -1446 | 1 | 9 | 0 | 251 | 285 | -4 | -5 | 1 | 633 | 626 | 7 | -3 | 1 | 168 | -195 |
| 5 | 2 | 0 | 87 | -123 | 0 | 5 | 0 | 452 | -464 | 2 | 9 | 0 | 203 | 229 | -2 | -5 | 1 | 286 | -289 | -6 | -2 | 1 | 269 | -290 |
| 7 | 2 | 0 | 398 | 379 | 1 | 5 | 0 | 204 | 225 | 3 | 9 | 0 | 289 | 315 | -1 | -5 | 1 | 508 | -498 | -4 | -2 | 1 | 261 | 240 |
| 8 | 2 | 0 | 261 | 292 | 2 | 5 | 0 | 54 | 51 | 0 | -9 | 1 | 221 | -253 | 0 | -5 | 1 | 381 | -368 | -3 | -2 | 1 | 1201 | 1129 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -2 | -2 | 1 | 1480 | 1418 | 7 | 0 | 1 | 300 | 322 | -1 | 3 | 1 | 323 | 337 | 0 | 6 | 1 | 441 | -456 | -2 | -8 | 2 | 307 | 281 |
| -1 | -2 | 1 | 154 | -275 | -8 | 1 | 1 | 310 | 316 | 0 | 3 | 1 | 267 | -108 | 3 | 6 | 1 | 478 | 481 | -1 | -8 | 2 | 240 | 228 |
| 0 | -2 | 1 | 270 | 287 | -7 | 1 | 1 | 488 | 491 | 1 | 3 | 1 | 1866 | 1941 | 4 | 6 | 1 | 375 | 385 | 0 | -8 | 2 | 204 | -187 |
| 1 | -2 | 1 | 516 | 587 | -6 | 1 | 1 | 577 | 568 | 2 | 3 | 1 | 1240 | 1196 | 5 | 6 | 1 | 383 | 398 | 1 | -8 | 2 | 239 | -254 |
| 2 | -2 | 1 | 1494 | -1582 | -5 | 1 | 1 | 90 | -66 | 3 | 3 | 1 | 112 | 77 | 6 | 6 | 1 | 349 | 355 | 2 | -8 | 2 | 161 | -165 |
| 3 | -2 | 1 | 927 | -937 | -4 | 1 | 1 | 260 | -257 | 4 | 3 | 1 | 58 | 49 | -5 | 7 | 1 | 163 | -184 | 4 | -8 | 2 | 121 | 129 |
| 4 | -2 | 1 | 157 | -142 | -3 | 1 | 1 | 245 | 350 | 5 | 3 | 1 | 148 | -160 | -2 | 7 | 1 | 172 | 145 | -5 | -7 | 2 | 235 | -259 |
| 5 | -2 | 1 | 79 | 36 | -2 | 1 | 1 | 760 | -707 | 7 | 3 | 1 | 91 | 55 | -1 | 7 | 1 | 359 | 365 | -4 | -7 | 2 | 316 | -340 |
| 6 | -2 | 1 | 217 | 237 | -1 | 1 | 1 | 484 | -495 | -6 | 4 | 1 | 139 | 155 | 0 | 7 | 1 | 463 | 475 | -3 | -7 | 2 | 301 | -300 |
| -8 | -1 | 1 | 167 | 180 | 0 | 1 | 1 | 648 | 783 | -5 | 4 | 1 | 136 | 167 | 1 | 7 | 1 | 546 | 563 | -2 | -7 | 2 | 302 | -294 |
| -7 | -1 | 1 | 243 | 240 | 1 | 1 | 1 | 1059 | 946 | -4 | 4 | 1 | 1011 | 991 | 2 | 7 | 1 | 208 | -203 | 1 | -7 | 2 | 111 | 161 |
| -5 | -1 | 1 | 105 | 112 | 2 | 1 | 1 | 443 | 407 | -3 | 4 | 1 | 936 | 928 | 3 | 7 | 1 | 345 | -375 | 2 | -7 | 2 | 333 | 350 |
| -4 | -1 | 1 | 122 | -122 | 4 | 1 | 1 | 639 | -650 | -2 | 4 | 1 | 204 | 144 | 4 | 7 | 1 | 127 | -159 | 3 | -7 | 2 | 236 | -235 |
| -3 | -1 | 1 | 1364 | -1370 | 5 | 1 | 1 | 333 | -344 | -1 | 4 | 1 | 130 | -67 | 5 | 7 | 1 | 347 | -349 | 4 | -7 | 2 | 418 | -404 |
| -2 | -1 | 1 | 1525 | -1404 | 6 | 1 | 1 | 645 | -651 | 0 | 4 | 1 | 407 | -401 | 6 | 7 | 1 | 136 | -147 | 5 | -7 | 2 | 172 | -211 |
| -1 | -1 | 1 | 611 | -527 | 7 | 1 | 1 | 433 | -442 | 1 | 4 | 1 | 652 | -626 | -4 | 8 | 1 | 225 | 227 | -4 | -6 | 2 | 225 | 250 |
| 0 | -1 | 1 | 119 | -58 | -7 | 2 | 1 | 390 | -392 | 2 | 4 | 1 | 348 | -214 | -3 | 8 | 1 | 168 | -133 | -3 | -6 | 2 | 644 | 654 |
| 1 | -1 | 1 | 724 | 727 | -6 | 2 | 1 | 393 | -376 | 3 | 4 | 1 | 437 | -413 | -2 | 8 | 1 | 197 | -193 | -2 | -6 | 2 | 137 | -141 |
| 2 | -1 | 1 | 277 | 212 | -5 | 2 | 1 | 245 | 257 | 4 | 4 | 1 | 461 | 435 | -1 | 8 | 1 | 198 | -204 | -1 | -6 | 2 | 398 | -401 |
| 3 | -1 | 1 | 296 | 253 | -4 | 2 | 1 | 267 | 225 | 5 | 4 | 1 | 381 | 355 | 0 | 8 | 1 | 604 | -637 | 1 | -6 | 2 | 202 | -259 |
| 4 | -1 | 1 | 539 | 529 | -3 | 2 | 1 | 76 | 39 | -5 | 5 | 1 | 210 | -217 | 1 | 8 | 1 | 408 | -440 | 2 | -6 | 2 | 185 | -188 |
| 5 | -1 | 1 | 578 | -560 | -2 | 2 | 1 | 50 | 26 | -4 | 5 | 1 | 538 | -554 | 2 | 8 | 1 | 134 | 79 | 3 | -6 | 2 | 282 | 268 |
| 6 | -1 | 1 | 503 | -477 | -1 | 2 | 1 | 539 | -607 | -3 | 5 | 1 | 491 | -497 | 4 | 8 | 1 | 164 | 198 | 4 | -6 | 2 | 406 | 413 |
| -8 | 0 | 1 | 365 | -364 | 0 | 2 | 1 | 824 | -744 | -2 | 5 | 1 | 536 | -539 | 5 | 8 | 1 | 101 | 100 | 5 | -6 | 2 | 498 | 504 |
| -7 | 0 | 1 | 414 | -414 | 1 | 2 | 1 | 1593 | -1582 | -1 | 5 | 1 | 288 | 282 | -2 | 9 | 1 | 204 | 224 | 6 | -6 | 2 | 238 | 217 |
| -6 | 0 | 1 | 211 | -214 | 2 | 2 | 1 | 1474 | -1515 | 0 | 5 | 1 | 517 | 495 | -1 | 9 | 1 | 243 | 256 | -4 | -5 | 2 | 241 | -252 |
| -5 | 0 | 1 | 352 | -361 | 3 | 2 | 1 | 93 | 143 | 1 | 5 | 1 | 491 | -516 | 0 | 9 | 1 | 275 | 258 | -3 | -5 | 2 | 322 | -321 |
| -4 | 0 | 1 | 228 | 247 | 4 | 2 | 1 | 232 | 231 | 2 | 5 | 1 | 59 | 34 | 1 | 9 | 1 | 118 | 138 | -2 | -5 | 2 | 433 | 425 |
| -3 | 0 | 1 | 663 | 628 | 6 | 2 | 1 | 518 | 520 | 3 | 5 | 1 | 123 | 19 | 2 | 9 | 1 | 101 | 99 | -1 | -5 | 2 | 543 | 524 |
| -2 | 0 | 1 | 1152 | 1052 | 7 | 2 | 1 | 213 | 220 | 4 | 5 | 1 | 883 | -882 | 3 | 9 | 1 | 114 | -157 | 0 | -5 | 2 | 851 | 848 |
| -1 | 0 | 1 | 889 | 938 | -7 | 3 | 1 | 149 | 168 | 5 | 5 | 1 | 454 | -466 | -1 | -9 | 2 | 93 | -78 | 1 | -5 | 2 | 480 | 483 |
| 1 | 0 | 1 | 946 | -869 | -6 | 3 | 1 | 142 | -153 | 6 | 5 | 1 | 173 | -193 | 0 | -9 | 2 | 219 | 243 | 2 | -5 | 2 | 146 | 148 |
| 2 | 0 | 1 | 930 | 998 | -5 | 3 | 1 | 230 | -254 | -5 | 6 | 1 | 147 | 146 | 1 | -9 | 2 | 432 | 445 | 3 | -5 | 2 | 131 | 129 |
| 3 | 0 | 1 | 228 | -212 | -4 | 3 | 1 | 771 | -782 | -3 | 6 | 1 | 311 | 287 | 2 | -9 | 2 | 164 | 209 | 4 | -5 | 2 | 331 | -357 |
| 5 | 0 | 1 | 910 | 927 | -3 | 3 | 1 | 965 | -967 | -2 | 6 | 1 | 267 | 255 | -4 | -8 | 2 | 371 | 358 | 5 | -5 | 2 | 645 | -597 |
| 6 | 0 | 1 | 625 | 648 | -2 | 3 | 1 | 263 | 269 | -1 | 6 | 1 | 684 | -683 | -3 | -8 | 2 | 189 | 218 | 6 | -5 | 2 | 139 | -130 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -6 | -4 | 2 | 119 | 142 | 4 | -2 | 2 | 272 | 192 | -2 | 1 | 2 | 1165 | 1021 | 3 | 3 | 2 | 97 | 49 | -1 | 6 | 2 | 263 | -223 |
| -5 | -4 | 2 | 247 | 270 | 5 | -2 | 2 | 181 | 187 | -1 | 1 | 2 | 2435 | 2616 | 4 | 3 | 2 | 96 | 78 | 0 | 6 | 2 | 339 | 357 |
| -4 | -4 | 2 | 136 | 150 | 7 | -2 | 2 | 265 | -308 | 0 | 1 | 2 | 297 | 254 | 5 | 3 | 2 | 152 | -45 | 1 | 6 | 2 | 190 | 70 |
| -3 | -4 | 2 | 98 | -86 | -6 | -1 | 2 | 125 | -150 | 1 | 1 | 2 | 233 | -251 | 6 | 3 | 2 | 486 | 480 | 2 | 6 | 2 | 525 | 490 |
| -2 | -4 | 2 | 72 | 97 | -5 | -1 | 2 | 437 | -446 | 2 | 1 | 2 | 337 | 432 | 7 | 3 | 2 | 404 | 403 | 3 | 6 | 2 | 415 | 404 |
| -1 | -4 | 2 | 904 | -868 | -4 | -1 | 2 | 972 | -979 | 3 | 1 | 2 | 640 | -616 | -7 | 4 | 2 | 181 | 224 | 4 | 6 | 2 | 85 | 31 |
| 0 | -4 | 2 | 1797 | -1844 | -2 | -1 | 2 | 755 | 777 | 4 | 1 | 2 | 575 | -553 | -6 | 4 | 2 | 480 | 473 | 6 | 6 | 2 | 212 | -252 |
| 1 | -4 | 2 | 371 | -329 | -1 | -1 | 2 | 524 | -653 | 5 | 1 | 2 | 136 | -119 | -5 | 4 | 2 | 614 | 588 | -5 | 7 | 2 | 214 | 225 |
| 2 | -4 | 2 | 199 | -191 | 0 | -1 | 2 | 180 | 165 | 6 | 1 | 2 | 92 | 84 | -3 | 4 | 2 | 534 | -519 | -4 | 7 | 2 | 149 | 185 |
| 3 | -4 | 2 | 217 | -236 | 1 | -1 | 2 | 339 | -294 | 7 | 1 | 2 | 383 | 379 | -2 | 4 | 2 | 347 | 353 | -3 | 7 | 2 | 494 | 486 |
| 4 | -4 | 2 | 300 | 283 | 2 | -1 | 2 | 716 | -759 | 8 | 1 | 2 | 90 | 117 | -1 | 4 | 2 | 160 | 171 | -2 | 7 | 2 | 633 | 650 |
| 5 | -4 | 2 | 278 | 310 | 3 | -1 | 2 | 1075 | -1071 | -7 | 2 | 2 | 309 | 311 | 0 | 4 | 2 | 260 | -53 | -1 | 7 | 2 | 430 | 437 |
| 7 | -4 | 2 | 126 | 12 | 4 | -1 | 2 | 978 | -1045 | -6 | 2 | 2 | 570 | 532 | 1 | 4 | 2 | 675 | 661 | 1 | 7 | 2 | 331 | -323 |
| -6 | -3 | 2 | 315 | -348 | 5 | -1 | 2 | 435 | -489 | -5 | 2 | 2 | 135 | 146 | 2 | 4 | 2 | 816 | 817 | 2 | 7 | 2 | 208 | -206 |
| -5 | -3 | 2 | 767 | -761 | 6 | -1 | 2 | 212 | 218 | -4 | 2 | 2 | 187 | -177 | 3 | 4 | 2 | 685 | 692 | 3 | 7 | 2 | 193 | 162 |
| -4 | -3 | 2 | 303 | -316 | 8 | -1 | 2 | 181 | 205 | -3 | 2 | 2 | 187 | 155 | 4 | 4 | 2 | 330 | 328 | 5 | 7 | 2 | 184 | 183 |
| -3 | -3 | 2 | 301 | -285 | -7 | 0 | 2 | 151 | 159 | -2 | 2 | 2 | 1251 | -1206 | 5 | 4 | 2 | 579 | -572 | 6 | 7 | 2 | 304 | 279 |
| -2 | -3 | 2 | 317 | -351 | -6 | 0 | 2 | 87 | -32 | -1 | 2 | 2 | 1508 | -1497 | 6 | 4 | 2 | 280 | -283 | -4 | 8 | 2 | 170 | -170 |
| -1 | -3 | 2 | 715 | 708 | -5 | 0 | 2 | 266 | 271 | 0 | 2 | 2 | 723 | -687 | 7 | 4 | 2 | 193 | -229 | -3 | 8 | 2 | 262 | -265 |
| 0 | -3 | 2 | 995 | 967 | -4 | 0 | 2 | 427 | 439 | 1 | 2 | 2 | 253 | -308 | -6 | 5 | 2 | 143 | -158 | -1 | 8 | 2 | 178 | -171 |
| 1 | -3 | 2 | 622 | 588 | -3 | 0 | 2 | 870 | -846 | 2 | 2 | 2 | 670 | 630 | -5 | 5 | 2 | 263 | -232 | 0 | 8 | 2 | 159 | 134 |
| 2 | -3 | 2 | 291 | 231 | -2 | 0 | 2 | 1067 | -938 | 3 | 2 | 2 | 190 | 170 | -4 | 5 | 2 | 216 | 203 | 1 | 8 | 2 | 435 | 449 |
| 3 | -3 | 2 | 408 | -400 | -1 | 0 | 2 | 1065 | -1034 | 4 | 2 | 2 | 280 | -303 | -3 | 5 | 2 | 552 | 512 | 2 | 8 | 2 | 69 | 108 |
| 5 | -3 | 2 | 90 | 48 | 0 | 0 | 2 | 976 | -1043 | 5 | 2 | 2 | 173 | 174 | -2 | 5 | 2 | 443 | 413 | 3 | 8 | 2 | 89 | -68 |
| 7 | -3 | 2 | 290 | 275 | 1 | 0 | 2 | 168 | -65 | 6 | 2 | 2 | 314 | -321 | -1 | 5 | 2 | 155 | 133 | 4 | 8 | 2 | 106 | -85 |
| -7 | -2 | 2 | 90 | -91 | 2 | 0 | 2 | 89 | 20 | 7 | 2 | 2 | 538 | -519 | 0 | 5 | 2 | 624 | -650 | 5 | 8 | 2 | 371 | -369 |
| -6 | -2 | 2 | 374 | 408 | 3 | 0 | 2 | 889 | 835 | 8 | 2 | 2 | 150 | -187 | 1 | 5 | 2 | 438 | -418 | 0 | 9 | 2 | 294 | -277 |
| -5 | -2 | 2 | 753 | 779 | 4 | 0 | 2 | 1180 | 1205 | -7 | 3 | 2 | 326 | -347 | 2 | 5 | 2 | 599 | -573 | 1 | 9 | 2 | 287 | -316 |
| -4 | -2 | 2 | 526 | 529 | 5 | 0 | 2 | 258 | 245 | -6 | 3 | 2 | 620 | -607 | 3 | 5 | 2 | 1041 | -1072 | 2 | 9 | 2 | 202 | -210 |
| -3 | -2 | 2 | 643 | 600 | 6 | 0 | 2 | 173 | -145 | -5 | 3 | 2 | 312 | -304 | 4 | 5 | 2 | 236 | -248 | -1 | -9 | 3 | 202 | 224 |
| -2 | -2 | 2 | 91 | -96 | 8 | 0 | 2 | 92 | -97 | -4 | 3 | 2 | 258 | -238 | 5 | 5 | 2 | 304 | 313 | 0 | -9 | 3 | 176 | 151 |
| -1 | -2 | 2 | 130 | 164 | -7 | 1 | 2 | 151 | -187 | -3 | 3 | 2 | 249 | 211 | 7 | 5 | 2 | 170 | 183 | 1 | -9 | 3 | 140 | -113 |
| 0 | -2 | 2 | 203 | -133 | -6 | 1 | 2 | 152 | -190 | -2 | 3 | 2 | 488 | 389 | -5 | 6 | 2 | 175 | -166 | -1 | -8 | 3 | 263 | -298 |
| 1 | -2 | 2 | 363 | -496 | -5 | 1 | 2 | 276 | -254 | 0 | 3 | 2 | 708 | 775 | -4 | 6 | 2 | 211 | -216 | 1 | -8 | 3 | 341 | 303 |
| 2 | -2 | 2 | 594 | 629 | -4 | 1 | 2 | 268 | 262 | 1 | 3 | 2 | 431 | -390 | -3 | 6 | 2 | 537 | -546 | 2 | -8 | 3 | 289 | 324 |
| 3 | -2 | 2 | 1127 | 1157 | -3 | 1 | 2 | 178 | 147 | 2 | 3 | 2 | 1443 | -1475 | -2 | 6 | 2 | 986 | -986 | 3 | -8 | 3 | 292 | 313 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -4 | -7 | 3 | 131 | 97 | 2 | -4 | 3 | 256 | -280 | -5 | -1 | 3 | 359 | 359 | 6 | 1 | 3 | 278 | 286 | -1 | 4 | 3 | 409 | 327 |
| -3 | -7 | 3 | 192 | 208 | 3 | -4 | 3 | 341 | 345 | -4 | -1 | 3 | 903 | 865 | 8 | 1 | 3 | 248 | -235 | 0 | 4 | 3 | 1619 | 1638 |
| -2 | -7 | 3 | 242 | 270 | 4 | -4 | 3 | 149 | -182 | -3 | -1 | 3 | 670 | 697 | -7 | 2 | 3 | 244 | 268 | 1 | 4 | 3 | 860 | 856 |
| -1 | -7 | 3 | 214 | 212 | 5 | -4 | 3 | 625 | -673 | -2 | -1 | 3 | 423 | -454 | -6 | 2 | 3 | 123 | -122 | 3 | 4 | 3 | 53 | -32 |
| 1 | -7 | 3 | 206 | -227 | 6 | -4 | 3 | 484 | -482 | -1 | -1 | 3 | 111 | 126 | -5 | 2 | 3 | 74 | 23 | 4 | 4 | 3 | 317 | -320 |
| 2 | -7 | 3 | 617 | -625 | 7 | -4 | 3 | 155 | -199 | 0 | -1 | 3 | 58 | 132 | -3 | 2 | 3 | 660 | -643 | 5 | 4 | 3 | 270 | -35 |
| 3 | -7 | 3 | 454 | -459 | -7 | -3 | 3 | 256 | -307 | 1 | -1 | 3 | 2770 | -2867 | -2 | 2 | 3 | 134 | -157 | 6 | 4 | 3 | 254 | 158 |
| 5 | -7 | 3 | 123 | -113 | -6 | -3 | 3 | 381 | -411 | 2 | -1 | 3 | 1091 | -1027 | -1 | 2 | 3 | 1567 | 1582 | 7 | 4 | 3 | 131 | 65 |
| -4 | -6 | 3 | 158 | -185 | -5 | -3 | 3 | 77 | 72 | 4 | -1 | 3 | 78 | -89 | 0 | 2 | 3 | 525 | 446 | -6 | 5 | 3 | 206 | 217 |
| -3 | -6 | 3 | 604 | -582 | -4 | -3 | 3 | 98 | 121 | 5 | -1 | 3 | 83 | 76 | 1 | 2 | 3 | 126 | 84 | -5 | 5 | 3 | 680 | 631 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 6 | 5 | 6 | 203 | 189 | 3 | -6 | 7 | 115 | -95 | 3 | -2 | 7 | 1223 | -1198 | 6 | 1 | 7 | 227 | -245 | 3 | 4 | 7 | 598 | -598 |
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| 1 | 6 | 6 | 188 | 188 | -1 | -5 | 7 | 554 | -528 | -5 | -1 | 7 | 135 | 188 | -4 | 2 | 7 | 372 | 304 | -5 | 5 | 7 | 120 | 51 |
| 2 | 6 | 6 | 100 | -125 | 0 | -5 | 7 | 616 | -588 | -4 | -1 | 7 | 455 | 421 | -2 | 2 | 7 | 427 | 392 | -4 | 5 | 7 | 291 | -322 |
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| 2 | 8 | 6 | 117 | -134 | 3 | -3 | 7 | 579 | 560 | -6 | 1 | 7 | 241 | 298 | 5 | 3 | 7 | 114 | -135 | 5 | 6 | 7 | 158 | 182 |
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| -1 | -6 | 7 | 433 | 388 | -1 | -2 | 7 | 176 | -78 | 2 | 1 | 7 | 145 | -173 | -1 | 4 | 7 | 273 | -230 | 3 | 7 | 7 | 254 | -227 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 3 | 8 | 7 | 103 | 111 | 0 | -2 | 8 | 419 | -421 | -3 | 2 | 8 | 156 | 130 | -4 | 5 | 8 | 359 | -387 | -2 | -5 | 9 | 359 | 322 |
| 4 | 8 | 7 | 136 | 135 | 1 | -2 | 8 | 899 | -867 | -1 | 2 | 8 | 959 | -934 | -3 | 5 | 8 | 154 | 154 | 0 | -5 | 9 | 163 | 167 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -4 | 0 | 9 | 286 | -295 | 1 | 4 | 9 | 869 | 940 | 3 | -5 | 10 | 155 | 157 | -5 | 1 | 10 | 435 | 399 | 4 | 4 | 10 | 224 | -216 |
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| 0 | 4 | 9 | 439 | 447 | 2 | -5 | 10 | 155 | -141 | 6 | 0 | 10 | 141 | 134 | 2 | 4 | 10 | 274 | -348 | 4 | -3 | 11 | 105 | -84 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 2 | -1 | 11 | 97 | -78 | -2 | 4 | 11 | 99 | -114 | -3 | -1 | 12 | 253 | 236 | -1 | 4 | 12 | 143 | 112 | -2 | 3 | 13 | 193 | -200 |
| 3 | -1 | 11 | 109 | -96 | -1 | 4 | 11 | 268 | -273 | -2 | -1 | 12 | 335 | 334 | 0 | 4 | 12 | 177 | 199 | -1 | 3 | 13 | 195 | -230 |
| 4 | -1 | 11 | 91 | -130 | 0 | 4 | 11 | 254 | -259 | -1 | -1 | 12 | 189 | 283 | 1 | 4 | 12 | 265 | 299 | 0 | 3 | 13 | 387 | -360 |
| 5 | -1 | 11 | 404 | -405 | 2 | 4 | 11 | 100 | 117 | 2 | -1 | 12 | 210 | -228 | 2 | 4 | 12 | 354 | 399 | 1 | 3 | 13 | 250 | -226 |
| 0 | 0 | 11 | 541 | -507 | 3 | 4 | 11 | 112 | 164 | 3 | -1 | 12 | 254 | -311 | 3 | 4 | 12 | 345 | 360 | 2 | 3 | 13 | 138 | 133 |
| 1 | 0 | 11 | 817 | -718 | 4 | 4 | 11 | 394 | 406 | 4 | -1 | 12 | 324 | -320 | -2 | 5 | 12 | 350 | 340 | 3 | 3 | 13 | 249 | 302 |
| 4 | 0 | 11 | 119 | 142 | 5 | 4 | 11 | 375 | 370 | -3 | 0 | 12 | 275 | -226 | 0 | 5 | 12 | 244 | -239 | -2 | 4 | 13 | 158 | 176 |
| 5 | 0 | 11 | 344 | 365 | -1 | 5 | 11 | 410 | 373 | -2 | 0 | 12 | 400 | -379 | 1 | 5 | 12 | 155 | -217 | 0 | 4 | 13 | 214 | 188 |
| -4 | 1 | 11 | 269 | -254 | 0 | 5 | 11 | 482 | 477 | -1 | 0 | 12 | 446 | -486 | 3 | 5 | 12 | 157 | -144 | 2 | 4 | 13 | 110 | -129 |
| 0 | 1 | 11 | 555 | 528 | 1 | 5 | 11 | 124 | -108 | 0 | 0 | 12 | 147 | -159 | 4 | 5 | 12 | 99 | 83 | 3 | 4 | 13 | 200 | -226 |
| 1 | 1 | 11 | 755 | 688 | 2 | 5 | 11 | 126 | -120 | 1 | 0 | 12 | 198 | 171 | 0 | 6 | 12 | 238 | 247 | -1 | 5 | 13 | 139 | -151 |
| 2 | 1 | 11 | 383 | 398 | 3 | 5 | 11 | 230 | -235 | 2 | 0 | 12 | 89 | 147 | 1 | 6 | 12 | 149 | 174 | 1 | 5 | 13 | 184 | 170 |
| 3 | 1 | 11 | 105 | -65 | 4 | 5 | 11 | 430 | -421 | 4 | 0 | 12 | 193 | 187 | 1 | 7 | 12 | 219 | -253 | 2 | 5 | 13 | 152 | 208 |
| 4 | 1 | 11 | 186 | -189 | 5 | 5 | 11 | 232 | -278 | -2 | 1 | 12 | 183 | 323 | 0 | -2 | 13 | 115 | 158 | 3 | 5 | 13 | 307 | 292 |
| -4 | 2 | 11 | 421 | 404 | -2 | 6 | 11 | 114 | -143 | -1 | 1 | 12 | 489 | 507 | 1 | -2 | 13 | 403 | 349 | 1 | 0 | 14 | 210 | -180 |
| -3 | 2 | 11 | 272 | 276 | -1 | 6 | 11 | 430 | -412 | 1 | 1 | 12 | 255 | -229 | 2 | -2 | 13 | 198 | 163 | 2 | 0 | 14 | 319 | -338 |
| -1 | 2 | 11 | 229 | -214 | 0 | 6 | 11 | 421 | -423 | 2 | 1 | 12 | 132 | -122 | 1 | -1 | 13 | 195 | -198 | 0 | 1 | 14 | 166 | 138 |
| 0 | 2 | 11 | 199 | -224 | 1 | 6 | 11 | 117 | -136 | 4 | 1 | 12 | 88 | 78 | 3 | -1 | 13 | 216 | 215 | 0 | 2 | 14 | 222 | -219 |
| 1 | 2 | 11 | 442 | -405 | 2 | 6 | 11 | 125 | 150 | -3 | 2 | 12 | 163 | -157 | -2 | 0 | 13 | 175 | 190 | 0 | 3 | 14 | 259 | 285 |
| 2 | 2 | 11 | 449 | -414 | 3 | 6 | 11 | 319 | 326 | -2 | 2 | 12 | 154 | -109 | -1 | 0 | 13 | 224 | 276 | 1 | 3 | 14 | 322 | 309 |
| 3 | 2 | 11 | 154 | 146 | 4 | 6 | 11 | 165 | 176 | -1 | 2 | 12 | 183 | -130 | 0 | 0 | 13 | 157 | 164 | 1 | 4 | 14 | 408 | -413 |
| 4 | 2 | 11 | 186 | 230 | -1 | 7 | 11 | 253 | 276 | 1 | 2 | 12 | 267 | 283 | | | | | | | | | | |

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR (2-N,N-dimethylbenzylamine-C,N') tellurium (IV) tribromide PAGE 1.

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 4 | 0 | 0 | 3341 | -3384 | 1 | 7 | 0 | 1060 | -1069 | 3 | 14 | 0 | 314 | -319 | 4 | 2 | 1 | 379 | -405 | -4 | 6 | 1 | 1556 | 1432 |
| 6 | 0 | 0 | 469 | 500 | 2 | 7 | 0 | 893 | 878 | 4 | 14 | 0 | 514 | 495 | 10 | 2 | 1 | 640 | -628 | -3 | 6 | 1 | 738 | -714 |
| 1 | 1 | 0 | 1175 | -1230 | 3 | 7 | 0 | 1263 | 1237 | 2 | 15 | 0 | 569 | 562 | -6 | 3 | 1 | 936 | -783 | -1 | 6 | 1 | 693 | -710 |
| 2 | 1 | 0 | 1345 | -1495 | 6 | 7 | 0 | 541 | -564 | 6 | 15 | 0 | 326 | -339 | -4 | 3 | 1 | 1227 | 1242 | 0 | 6 | 1 | 1934 | -1986 |
| 3 | 1 | 0 | 1205 | 1323 | 7 | 7 | 0 | 532 | -529 | 0 | 16 | 0 | 772 | 780 | -2 | 3 | 1 | 834 | 814 | 1 | 6 | 1 | 411 | 389 |
| 6 | 1 | 0 | 530 | 543 | 10 | 7 | 0 | 342 | 345 | 1 | 16 | 0 | 462 | -460 | -1 | 3 | 1 | 1209 | -1166 | 2 | 6 | 1 | 717 | -683 |
| 7 | 1 | 0 | 1052 | -1007 | 0 | 8 | 0 | 3116 | 3129 | 3 | 16 | 0 | 496 | -478 | 0 | 3 | 1 | 1736 | -1761 | 4 | 6 | 1 | 305 | 296 |
| 1 | 2 | 0 | 1404 | 1417 | 1 | 8 | 0 | 422 | -465 | 4 | 16 | 0 | 482 | -470 | 3 | 3 | 1 | 1160 | 1082 | 6 | 6 | 1 | 371 | -364 |
| 2 | 2 | 0 | 263 | 257 | 3 | 8 | 0 | 1005 | -989 | 1 | 17 | 0 | 482 | -462 | 4 | 3 | 1 | 850 | 808 | 7 | 6 | 1 | 405 | 428 |
| 3 | 2 | 0 | 2397 | 2537 | 4 | 8 | 0 | 1624 | -1626 | 3 | 17 | 0 | 471 | 456 | 7 | 3 | 1 | 594 | -617 | 10 | 6 | 1 | 478 | 522 |
| 5 | 2 | 0 | 441 | 447 | 6 | 8 | 0 | 270 | 267 | 0 | 18 | 0 | 531 | 538 | -7 | 4 | 1 | 2095 | 1809 | -6 | 7 | 1 | 790 | 737 |
| 7 | 2 | 0 | 656 | -716 | 7 | 8 | 0 | 335 | 342 | -7 | 0 | 1 | 1790 | -2052 | -4 | 4 | 1 | 620 | -596 | -5 | 7 | 1 | 330 | -321 |
| 1 | 3 | 0 | 1149 | 1110 | 1 | 9 | 0 | 875 | -891 | -3 | 0 | 1 | 4118 | 4238 | -3 | 4 | 1 | 3513 | -3443 | -4 | 7 | 1 | 962 | -896 |
| 2 | 3 | 0 | 557 | -566 | 2 | 9 | 0 | 220 | -221 | 1 | 0 | 1 | 1716 | -1775 | -1 | 4 | 1 | 567 | -532 | -3 | 7 | 1 | 232 | 239 |
| 3 | 3 | 0 | 1176 | -1202 | 3 | 9 | 0 | 1088 | 1094 | 3 | 0 | 1 | 1774 | 1805 | 0 | 4 | 1 | 721 | 755 | -2 | 7 | 1 | 974 | -924 |
| 4 | 3 | 0 | 203 | 195 | 4 | 9 | 0 | 353 | -374 | 5 | 0 | 1 | 843 | -857 | 1 | 4 | 1 | 1130 | 1109 | -1 | 7 | 1 | 1066 | 1071 |
| 6 | 3 | 0 | 375 | 397 | 7 | 9 | 0 | 777 | -797 | 7 | 0 | 1 | 1556 | -1516 | 2 | 4 | 1 | 208 | 221 | 0 | 7 | 1 | 1219 | 1218 |
| 7 | 3 | 0 | 715 | 755 | 0 | 10 | 0 | 1210 | 1233 | -10 | 1 | 1 | 379 | -510 | 3 | 4 | 1 | 754 | -695 | 1 | 7 | 1 | 215 | -191 |
| 0 | 4 | 0 | 4948 | -5204 | 1 | 10 | 0 | 519 | 510 | -6 | 1 | 1 | 579 | 662 | 5 | 4 | 1 | 710 | 685 | 3 | 7 | 1 | 1122 | -1097 |
| 1 | 4 | 0 | 454 | 456 | 2 | 10 | 0 | 313 | 320 | -4 | 1 | 1 | 1461 | -1462 | 7 | 4 | 1 | 1034 | 1030 | 4 | 7 | 1 | 624 | -592 |
| 2 | 4 | 0 | 178 | -176 | 3 | 10 | 0 | 884 | 869 | -2 | 1 | 1 | 337 | -420 | -10 | 5 | 1 | 453 | 474 | 6 | 7 | 1 | 512 | 525 |
| 3 | 4 | 0 | 816 | 817 | 4 | 10 | 0 | 634 | -611 | -1 | 1 | 1 | 1446 | -1507 | -6 | 5 | 1 | 682 | -609 | 7 | 7 | 1 | 595 | 612 |
| 4 | 4 | 0 | 2287 | 2280 | 6 | 10 | 0 | 325 | -317 | 0 | 1 | 1 | 1903 | 2043 | -4 | 5 | 1 | 1460 | 1371 | -7 | 8 | 1 | 1338 | -1268 |
| 6 | 4 | 0 | 286 | -271 | 1 | 11 | 0 | 655 | 664 | 1 | 1 | 1 | 495 | 463 | -3 | 5 | 1 | 263 | -245 | -6 | 8 | 1 | 347 | 331 |
| 10 | 4 | 0 | 411 | 425 | 2 | 11 | 0 | 618 | -619 | 3 | 1 | 1 | 1368 | 1332 | -2 | 5 | 1 | 697 | 642 | -4 | 8 | 1 | 809 | 783 |
| 1 | 5 | 0 | 1277 | 1256 | 3 | 11 | 0 | 622 | -601 | 4 | 1 | 1 | 1138 | -1099 | -1 | 5 | 1 | 570 | 548 | -3 | 8 | 1 | 2414 | 2329 |
| 3 | 5 | 0 | 1588 | -1616 | 6 | 11 | 0 | 396 | 435 | 5 | 1 | 1 | 437 | -432 | 0 | 5 | 1 | 2097 | -2141 | 0 | 8 | 1 | 702 | -744 |
| 4 | 5 | 0 | 283 | 294 | 0 | 12 | 0 | 1543 | -1555 | 7 | 1 | 1 | 590 | -558 | 1 | 5 | 1 | 883 | 874 | 1 | 8 | 1 | 1019 | -1027 |
| 7 | 5 | 0 | 1066 | 1086 | 1 | 12 | 0 | 558 | 561 | -6 | 2 | 1 | 1141 | -1069 | 3 | 5 | 1 | 469 | -444 | 2 | 8 | 1 | 425 | -435 |
| 0 | 6 | 0 | 1631 | -1603 | 3 | 12 | 0 | 747 | 730 | -4 | 2 | 1 | 1540 | -1653 | 4 | 5 | 1 | 1395 | 1328 | 3 | 8 | 1 | 732 | 724 |
| 1 | 6 | 0 | 1291 | -1240 | 4 | 12 | 0 | 895 | 874 | -3 | 2 | 1 | 246 | 257 | 5 | 5 | 1 | 318 | -311 | 7 | 8 | 1 | 846 | -906 |
| 2 | 6 | 0 | 453 | -438 | 6 | 12 | 0 | 293 | -287 | -2 | 2 | 1 | 265 | -275 | 7 | 5 | 1 | 363 | 356 | -6 | 9 | 1 | 584 | 540 |
| 3 | 6 | 0 | 1684 | -1690 | 1 | 13 | 0 | 615 | 612 | -1 | 2 | 1 | 677 | 682 | 8 | 5 | 1 | 402 | -415 | -4 | 9 | 1 | 1036 | -1006 |
| 4 | 6 | 0 | 750 | 741 | 3 | 13 | 0 | 761 | -735 | 0 | 2 | 1 | 1902 | 1913 | -7 | 6 | 1 | 459 | 395 | -2 | 9 | 1 | 601 | -580 |
| 6 | 6 | 0 | 376 | 373 | 7 | 13 | 0 | 561 | 551 | 2 | 2 | 1 | 2063 | 2011 | -6 | 6 | 1 | 844 | 769 | 0 | 9 | 1 | 1388 | 1452 |
| 7 | 6 | 0 | 443 | 451 | 0 | 14 | 0 | 870 | -904 | 3 | 2 | 1 | 376 | -375 | -5 | 6 | 1 | 289 | 261 | 1 | 9 | 1 | 555 | -556 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2 | 9 | 1 | 268 | 265 | -4 | 14 | 1 | 359 | 358 | -1 | 2 | 2 | 1152 | 1139 | 7 | 5 | 2 | 522 | 558 | -1 | 9 | 2 | 291 | 286 |
| 4 | 9 | 1 | 943 | -923 | -3 | 14 | 1 | 640 | -650 | 0 | 2 | 2 | 265 | 258 | -7 | 6 | 2 | 709 | 780 | 1 | 9 | 2 | 676 | -661 |
| -7 | 10 | 1 | 452 | -427 | -1 | 14 | 1 | 390 | -417 | 3 | 2 | 2 | 1391 | 1368 | -5 | 6 | 2 | 331 | 325 | 3 | 9 | 2 | 491 | 483 |
| -6 | 10 | 1 | 363 | -353 | 0 | 14 | 1 | 581 | -596 | 7 | 2 | 2 | 1631 | -1644 | -4 | 6 | 2 | 457 | -438 | 4 | 9 | 2 | 351 | -339 |
| -4 | 10 | 1 | 810 | -786 | 1 | 14 | 1 | 308 | 309 | -7 | 3 | 2 | 876 | 917 | -3 | 6 | 2 | 926 | -915 | 6 | 9 | 2 | 339 | 370 |
| -3 | 10 | 1 | 775 | 774 | -5 | 15 | 1 | 389 | -379 | -5 | 3 | 2 | 332 | 398 | -1 | 6 | 2 | 261 | -260 | 7 | 9 | 2 | 389 | -442 |
| -1 | 10 | 1 | 728 | 759 | -1 | 15 | 1 | 625 | 669 | -4 | 3 | 2 | 1041 | -1294 | 0 | 6 | 2 | 346 | -355 | -7 | 10 | 2 | 578 | -580 |
| 0 | 10 | 1 | 1117 | 1145 | 3 | 15 | 1 | 534 | -530 | -3 | 3 | 2 | 1260 | -1442 | 1 | 6 | 2 | 209 | -204 | -4 | 10 | 2 | 362 | 343 |
| 1 | 10 | 1 | 347 | -340 | -4 | 16 | 1 | 455 | 447 | -2 | 3 | 2 | 613 | -599 | 2 | 6 | 2 | 391 | 375 | -3 | 10 | 2 | 779 | 773 |
| 4 | 10 | 1 | 324 | -304 | -3 | 16 | 1 | 685 | 698 | -1 | 3 | 2 | 421 | 412 | 3 | 6 | 2 | 1466 | -1426 | 0 | 10 | 2 | 289 | 298 |
| 6 | 10 | 1 | 355 | 378 | 0 | 16 | 1 | 403 | -381 | 0 | 3 | 2 | 1533 | 1518 | 4 | 6 | 2 | 861 | 822 | 3 | 10 | 2 | 945 | 943 |
| 7 | 10 | 1 | 345 | -350 | 1 | 16 | 1 | 529 | -526 | 1 | 3 | 2 | 461 | 466 | 5 | 6 | 2 | 343 | 348 | 4 | 10 | 2 | 770 | -767 |
| -6 | 11 | 1 | 427 | -440 | 3 | 16 | 1 | 434 | 433 | 3 | 3 | 2 | 965 | -943 | 7 | 6 | 2 | 1246 | 1304 | 7 | 10 | 2 | 735 | -784 |
| -5 | 11 | 1 | 351 | 353 | -4 | 17 | 1 | 392 | -390 | 4 | 3 | 2 | 703 | -680 | -7 | 7 | 2 | 504 | -507 | -4 | 11 | 2 | 686 | -686 |
| -4 | 11 | 1 | 407 | 400 | -2 | 17 | 1 | 330 | -352 | 6 | 3 | 2 | 470 | 469 | -4 | 7 | 2 | 1252 | 1223 | -1 | 11 | 2 | 361 | 372 |
| -2 | 11 | 1 | 490 | 493 | 0 | 17 | 1 | 481 | 503 | -10 | 4 | 2 | 830 | 785 | -3 | 7 | 2 | 756 | 711 | 0 | 11 | 2 | 749 | 756 |
| -1 | 11 | 1 | 845 | -876 | -10 | 0 | 2 | 691 | -917 | -6 | 4 | 2 | 949 | -1125 | -2 | 7 | 2 | 475 | 451 | 3 | 11 | 2 | 566 | -569 |
| 0 | 11 | 1 | 422 | -436 | -6 | 0 | 2 | 1186 | 1252 | -2 | 4 | 2 | 386 | -354 | -1 | 7 | 2 | 562 | -560 | 4 | 11 | 2 | 410 | -419 |
| 3 | 11 | 1 | 789 | 799 | -2 | 0 | 2 | 547 | 429 | 0 | 4 | 2 | 1627 | -1630 | 0 | 7 | 2 | 1172 | -1185 | 6 | 11 | 2 | 528 | 555 |
| 6 | 11 | 1 | 354 | -370 | 0 | 0 | 2 | 1602 | 1649 | 1 | 4 | 2 | 502 | 483 | 1 | 7 | 2 | 530 | -511 | -7 | 12 | 2 | 459 | -449 |
| 7 | 11 | 1 | 390 | -403 | 2 | 0 | 2 | 1396 | -1380 | 2 | 4 | 2 | 1101 | 1031 | 3 | 7 | 2 | 909 | 893 | -6 | 12 | 2 | 643 | -627 |
| -7 | 12 | 1 | 684 | 693 | 4 | 0 | 2 | 2647 | -2606 | 3 | 4 | 2 | 280 | 262 | 4 | 7 | 2 | 519 | 503 | -4 | 12 | 2 | 318 | 330 |
| -6 | 12 | 1 | 415 | -405 | 8 | 0 | 2 | 768 | 847 | 4 | 4 | 2 | 2060 | 1987 | 6 | 7 | 2 | 610 | -627 | 0 | 12 | 2 | 828 | -850 |
| -4 | 12 | 1 | 786 | -771 | -7 | 1 | 2 | 1066 | -1161 | 5 | 4 | 2 | 388 | -371 | -7 | 8 | 2 | 577 | 536 | 1 | 12 | 2 | 532 | 563 |
| -3 | 12 | 1 | 1352 | -1332 | -5 | 1 | 2 | 392 | -465 | 8 | 4 | 2 | 486 | -556 | -6 | 8 | 2 | 1062 | 1061 | 3 | 12 | 2 | 408 | 419 |
| 0 | 12 | 1 | 660 | 692 | -4 | 1 | 2 | 833 | -967 | -7 | 5 | 2 | 963 | 1092 | -3 | 8 | 2 | 257 | -220 | 4 | 12 | 2 | 822 | 847 |
| 1 | 12 | 1 | 793 | 797 | -3 | 1 | 2 | 1675 | 1710 | -5 | 5 | 2 | 411 | 470 | 0 | 8 | 2 | 1255 | 1309 | 5 | 12 | 2 | 366 | -365 |
| 3 | 12 | 1 | 545 | -550 | -2 | 1 | 2 | 868 | -791 | -4 | 5 | 2 | 313 | 306 | 1 | 8 | 2 | 649 | -663 | -7 | 13 | 2 | 532 | 534 |
| 7 | 12 | 1 | 532 | 595 | 0 | 1 | 2 | 1066 | 1073 | -3 | 5 | 2 | 1643 | -1619 | 2 | 8 | 2 | 674 | -634 | -5 | 13 | 2 | 365 | 366 |
| -6 | 13 | 1 | 502 | -497 | 1 | 1 | 2 | 371 | -378 | -2 | 5 | 2 | 430 | 420 | 3 | 8 | 2 | 280 | -283 | -3 | 13 | 2 | 895 | -874 |
| -4 | 13 | 1 | 747 | 705 | 3 | 1 | 2 | 612 | 618 | -1 | 5 | 2 | 383 | -353 | 4 | 8 | 2 | 1446 | -1462 | 1 | 13 | 2 | 635 | 619 |
| -2 | 13 | 1 | 580 | 587 | 4 | 1 | 2 | 507 | -501 | 0 | 5 | 2 | 459 | -465 | 5 | 8 | 2 | 426 | 405 | 3 | 13 | 2 | 410 | -412 |
| 0 | 13 | 1 | 881 | -894 | 6 | 1 | 2 | 705 | 708 | 1 | 5 | 2 | 801 | 761 | -7 | 9 | 2 | 864 | -798 | -3 | 14 | 2 | 438 | -449 |
| 1 | 13 | 1 | 389 | 383 | -7 | 2 | 2 | 1036 | -1066 | 3 | 5 | 2 | 574 | -549 | -5 | 9 | 2 | 545 | -534 | 3 | 14 | 2 | 604 | -599 |
| 4 | 13 | 1 | 587 | 569 | -5 | 2 | 2 | 495 | -602 | 4 | 5 | 2 | 607 | 580 | -3 | 9 | 2 | 1287 | 1229 | 4 | 14 | 2 | 591 | 595 |
| -7 | 14 | 1 | 396 | 389 | -3 | 2 | 2 | 1012 | 1188 | 6 | 5 | 2 | 409 | -397 | -2 | 9 | 2 | 314 | -299 | -4 | 15 | 2 | 527 | 549 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0 | 15 | 2 | 485 | -497 | -10 | 3 | 3 | 491 | 430 | -3 | 6 | 3 | 872 | -841 | 2 | 9 | 3 | 444 | 457 | 0 | 14 | 3 | 713 | -741 |
| 2 | 15 | 2 | 357 | 326 | -7 | 3 | 3 | 698 | -781 | -2 | 6 | 3 | 503 | -470 | 3 | 9 | 3 | 720 | 704 | 1 | 14 | 3 | 623 | 628 |
| -2 | 16 | 2 | 354 | -359 | -6 | 3 | 3 | 556 | -624 | 0 | 6 | 3 | 2353 | -2406 | 4 | 9 | 3 | 857 | -863 | 4 | 14 | 3 | 620 | 644 |
| 0 | 16 | 2 | 485 | 483 | -5 | 3 | 3 | 430 | -498 | 1 | 6 | 3 | 953 | 926 | 5 | 9 | 3 | 367 | 361 | -3 | 15 | 3 | 342 | -326 |
| 4 | 16 | 2 | 441 | -457 | -4 | 3 | 3 | 268 | 296 | 2 | 6 | 3 | 590 | 547 | -6 | 10 | 3 | 355 | 335 | -1 | 15 | 3 | 451 | 475 |
| -3 | 17 | 2 | 393 | 395 | -3 | 3 | 3 | 650 | 646 | 3 | 6 | 3 | 442 | 413 | -5 | 10 | 3 | 332 | 307 | 3 | 15 | 3 | 754 | -758 |
| 1 | 17 | 2 | 357 | -330 | -2 | 3 | 3 | 328 | 343 | 4 | 6 | 3 | 2123 | 2032 | -4 | 10 | 3 | 562 | -533 | -3 | 16 | 3 | 379 | 380 |
| -7 | 0 | 3 | 704 | -648 | -1 | 3 | 3 | 576 | -542 | 8 | 6 | 3 | 482 | -503 | -3 | 10 | 3 | 544 | 522 | 0 | 16 | 3 | 448 | -473 |
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| -3 | 0 | 3 | 2824 | 2385 | 1 | 3 | 3 | 368 | -377 | -6 | 7 | 3 | 341 | 296 | 1 | 10 | 3 | 714 | -709 | 0 | 17 | 3 | 354 | 359 |
| -1 | 0 | 3 | 332 | -300 | 2 | 3 | 3 | 331 | -289 | -5 | 7 | 3 | 295 | 250 | 2 | 10 | 3 | 310 | -291 | -6 | 0 | 4 | 1436 | 1399 |
| 1 | 0 | 3 | 2939 | -2978 | 3 | 3 | 3 | 1847 | 1755 | -4 | 7 | 3 | 451 | -445 | 3 | 10 | 3 | 464 | -433 | -2 | 0 | 4 | 1729 | -1578 |
| 3 | 0 | 3 | 615 | -578 | 4 | 3 | 3 | 647 | 642 | -3 | 7 | 3 | 759 | -750 | 4 | 10 | 3 | 1248 | -1239 | 0 | 0 | 4 | 652 | -718 |
| 5 | 0 | 3 | 791 | 780 | 7 | 3 | 3 | 1042 | -1076 | -1 | 7 | 3 | 648 | 662 | -7 | 11 | 3 | 492 | -462 | -6 | 1 | 4 | 310 | 306 |
| -10 | 1 | 3 | 568 | -560 | -7 | 4 | 3 | 491 | 544 | 0 | 7 | 3 | 832 | 857 | -4 | 11 | 3 | 354 | 344 | -4 | 1 | 4 | 1027 | -951 |
| -7 | 1 | 3 | 606 | -631 | -5 | 4 | 3 | 458 | -511 | 1 | 7 | 3 | 324 | 324 | -3 | 11 | 3 | 323 | 289 | -3 | 1 | 4 | 1551 | 1386 |
| -6 | 1 | 3 | 760 | 707 | -3 | 4 | 3 | 1868 | -1881 | 3 | 7 | 3 | 1594 | -1557 | -1 | 11 | 3 | 385 | -397 | -2 | 1 | 4 | 505 | -464 |
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| -3 | 1 | 3 | 756 | 688 | -1 | 4 | 3 | 656 | 620 | 7 | 7 | 3 | 889 | 928 | 3 | 11 | 3 | 1058 | 1052 | 0 | 1 | 4 | 2274 | 2425 |
| -2 | 1 | 3 | 239 | -203 | 0 | 4 | 3 | 423 | 464 | -7 | 8 | 3 | 473 | -443 | 7 | 11 | 3 | 574 | -674 | 1 | 1 | 4 | 1067 | -1082 |
| -1 | 1 | 3 | 282 | -262 | 1 | 4 | 3 | 2481 | 2386 | -4 | 8 | 3 | 465 | 470 | -4 | 12 | 3 | 418 | -437 | 2 | 1 | 4 | 705 | 662 |
| 0 | 1 | 3 | 1412 | 1444 | 3 | 4 | 3 | 434 | 403 | -3 | 8 | 3 | 1409 | 1367 | -3 | 12 | 3 | 647 | -648 | 3 | 1 | 4 | 861 | -808 |
| 1 | 1 | 3 | 862 | -842 | 4 | 4 | 3 | 441 | -418 | -2 | 8 | 3 | 759 | -720 | -2 | 12 | 3 | 623 | 618 | 4 | 1 | 4 | 1548 | -1499 |
| 2 | 1 | 3 | 364 | 366 | 5 | 4 | 3 | 589 | -613 | -1 | 8 | 3 | 312 | -318 | -1 | 12 | 3 | 349 | 360 | -7 | 2 | 4 | 1150 | -1167 |
| 3 | 1 | 3 | 1581 | 1513 | -10 | 5 | 3 | 557 | 546 | 0 | 8 | 3 | 877 | -896 | 0 | 12 | 3 | 717 | 748 | -3 | 2 | 4 | 3084 | 2987 |
| 4 | 1 | 3 | 909 | -902 | -6 | 5 | 3 | 590 | -664 | 1 | 8 | 3 | 1557 | -1556 | 1 | 12 | 3 | 692 | 701 | -1 | 2 | 4 | 610 | -628 |
| 5 | 1 | 3 | 408 | 423 | -5 | 5 | 3 | 381 | 381 | 2 | 8 | 3 | 282 | 251 | 2 | 12 | 3 | 425 | -417 | 1 | 2 | 4 | 2455 | -2555 |
| 7 | 1 | 3 | 1027 | -1028 | -3 | 5 | 3 | 291 | -290 | 4 | 8 | 3 | 588 | 568 | 4 | 12 | 3 | 487 | -453 | 3 | 2 | 4 | 922 | 886 |
| -4 | 2 | 3 | 941 | -961 | 0 | 5 | 3 | 1464 | -1472 | 5 | 8 | 3 | 368 | 408 | -6 | 13 | 3 | 516 | -509 | 7 | 2 | 4 | 998 | -922 |
| -3 | 2 | 3 | 374 | 376 | 1 | 5 | 3 | 649 | 645 | -8 | 9 | 3 | 360 | -319 | -2 | 13 | 3 | 442 | 446 | -4 | 3 | 4 | 964 | -999 |
| -2 | 2 | 3 | 708 | 709 | 2 | 5 | 3 | 359 | -351 | -6 | 9 | 3 | 736 | 706 | 0 | 13 | 3 | 781 | -831 | -3 | 3 | 4 | 1062 | -1064 |
| 0 | 2 | 3 | 3389 | 3707 | 3 | 5 | 3 | 1123 | -1038 | -3 | 9 | 3 | 491 | 450 | 1 | 13 | 3 | 305 | 322 | -2 | 3 | 4 | 277 | -254 |
| 1 | 2 | 3 | 536 | -507 | 4 | 5 | 3 | 1109 | 1062 | -2 | 9 | 3 | 487 | -479 | 2 | 13 | 3 | 434 | -416 | -1 | 3 | 4 | 929 | -899 |
| 2 | 2 | 3 | 665 | -647 | 5 | 5 | 3 | 547 | -532 | -1 | 9 | 3 | 415 | -422 | 4 | 13 | 3 | 643 | 649 | 0 | 3 | 4 | 2285 | 2427 |
| 4 | 2 | 3 | 2804 | -2752 | 7 | 5 | 3 | 694 | 758 | 0 | 9 | 3 | 1063 | 1091 | -4 | 14 | 3 | 358 | 333 | 1 | 3 | 4 | 848 | 850 |
| 8 | 2 | 3 | 652 | 652 | -4 | 6 | 3 | 650 | 639 | 1 | 9 | 3 | 628 | -628 | -3 | 14 | 3 | 429 | -433 | 2 | 3 | 4 | 495 | 479 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 | 3 | 4 | 526 | 522 | -2 | 8 | 4 | 1144 | -1109 | 3 | 13 | 4 | 438 | 435 | -7 | 3 | 5 | 1025 | -1011 | -5 | 8 | 5 | 376 | -384 |
| 4 | 3 | 4 | 1535 | -1467 | 0 | 8 | 4 | 436 | -451 | -6 | 14 | 4 | 392 | -357 | -6 | 3 | 5 | 573 | -511 | -3 | 8 | 5 | 327 | -300 |
| -10 | 4 | 4 | 464 | 478 | 1 | 8 | 4 | 450 | 477 | -3 | 14 | 4 | 778 | -767 | -5 | 3 | 5 | 400 | -346 | -6 | 9 | 5 | 595 | 550 |
| -6 | 4 | 4 | 1063 | -1160 | 2 | 8 | 4 | 365 | 354 | -2 | 14 | 4 | 417 | 414 | -4 | 3 | 5 | 628 | -606 | -4 | 9 | 5 | 806 | 780 |
| -2 | 4 | 4 | 1174 | 1185 | -6 | 9 | 4 | 455 | 446 | 0 | 14 | 4 | 367 | 386 | -3 | 3 | 5 | 2003 | 1993 | -3 | 9 | 5 | 637 | 615 |
| -1 | 4 | 4 | 272 | 260 | -4 | 9 | 4 | 549 | -539 | 1 | 14 | 4 | 753 | 761 | -2 | 3 | 5 | 256 | 227 | 0 | 9 | 5 | 738 | -738 |
| 0 | 4 | 4 | 719 | 728 | -3 | 9 | 4 | 1191 | 1153 | 3 | 14 | 4 | 459 | -445 | -1 | 3 | 5 | 730 | 771 | 1 | 9 | 5 | 479 | -507 |
| -6 | 5 | 4 | 352 | -380 | -2 | 9 | 4 | 668 | -662 | -4 | 15 | 4 | 554 | 558 | 0 | 3 | 5 | 322 | 351 | 3 | 9 | 5 | 680 | 633 |
| -4 | 5 | 4 | 564 | 562 | -1 | 9 | 4 | 777 | 813 | 0 | 15 | 4 | 853 | -876 | 1 | 3 | 5 | 897 | -941 | -6 | 10 | 5 | 814 | 775 |
| -3 | 5 | 4 | 1334 | -1338 | 0 | 9 | 4 | 953 | 964 | 4 | 15 | 4 | 549 | 565 | 7 | 3 | 5 | 605 | -564 | -4 | 10 | 5 | 417 | -418 |
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| 0 | 5 | 4 | 1340 | -1359 | 4 | 9 | 4 | 540 | -571 | -1 | 17 | 4 | 362 | 395 | -3 | 4 | 5 | 471 | 452 | 4 | 10 | 5 | 891 | -845 |
| 1 | 5 | 4 | 904 | 887 | -7 | 10 | 4 | 626 | -576 | -9 | 0 | 5 | 439 | 472 | 1 | 4 | 5 | 344 | -325 | -7 | 11 | 5 | 629 | -612 |
| 2 | 5 | 4 | 673 | -612 | -6 | 10 | 4 | 377 | 351 | -5 | 0 | 5 | 457 | -456 | 7 | 4 | 5 | 518 | -477 | -3 | 11 | 5 | 1079 | 1048 |
| 3 | 5 | 4 | 865 | 775 | -3 | 10 | 4 | 1506 | 1459 | -3 | 0 | 5 | 586 | -504 | -7 | 5 | 5 | 566 | 586 | -1 | 11 | 5 | 508 | 523 |
| 4 | 5 | 4 | 974 | 931 | -1 | 10 | 4 | 454 | -466 | 7 | 0 | 5 | 638 | 583 | -6 | 5 | 5 | 722 | -720 | 1 | 11 | 5 | 587 | -596 |
| -7 | 6 | 4 | 831 | 904 | 0 | 10 | 4 | 527 | -544 | -7 | 1 | 5 | 909 | -913 | -5 | 5 | 5 | 320 | 286 | -6 | 13 | 5 | 346 | -315 |
| -6 | 6 | 4 | 382 | -420 | 1 | 10 | 4 | 1390 | -1402 | -6 | 1 | 5 | 744 | 706 | -4 | 5 | 5 | 1022 | -1012 | -4 | 13 | 5 | 595 | -616 |
| -4 | 6 | 4 | 346 | -342 | 3 | 10 | 4 | 626 | 603 | -5 | 1 | 5 | 437 | -389 | -3 | 5 | 5 | 1176 | -1138 | 0 | 13 | 5 | 565 | 558 |
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| -2 | 6 | 4 | 520 | 508 | 7 | 10 | 4 | 598 | -597 | -3 | 1 | 5 | 1989 | 1769 | 1 | 5 | 5 | 606 | 608 | 0 | 14 | 5 | 462 | -495 |
| -1 | 6 | 4 | 403 | 413 | -4 | 11 | 4 | 644 | -638 | -1 | 1 | 5 | 426 | 434 | 2 | 5 | 5 | 565 | 518 | 4 | 14 | 5 | 496 | 514 |
| 0 | 6 | 4 | 416 | 423 | 0 | 11 | 4 | 1152 | 1198 | 0 | 1 | 5 | 822 | -854 | 3 | 5 | 5 | 463 | -422 | -3 | 15 | 5 | 676 | -691 |
| 1 | 6 | 4 | 1923 | 1928 | 2 | 11 | 4 | 485 | 503 | 1 | 1 | 5 | 802 | -839 | -6 | 6 | 5 | 1211 | -1217 | 1 | 15 | 5 | 461 | 462 |
| 3 | 6 | 4 | 692 | -647 | 4 | 11 | 4 | 775 | -765 | 2 | 1 | 5 | 482 | -472 | -4 | 6 | 5 | 410 | 420 | -4 | 0 | 6 | 350 | 313 |
| 7 | 6 | 4 | 811 | 770 | -7 | 12 | 4 | 426 | -416 | 3 | 1 | 5 | 447 | 436 | -2 | 6 | 5 | 1214 | 1171 | 0 | 0 | 6 | 457 | -505 |
| -4 | 7 | 4 | 1046 | 1046 | -6 | 12 | 4 | 471 | -445 | 7 | 1 | 5 | 635 | -584 | 0 | 6 | 5 | 925 | -938 | 2 | 0 | 6 | 457 | 448 |
| -3 | 7 | 4 | 486 | 456 | -3 | 12 | 4 | 744 | 709 | -10 | 2 | 5 | 633 | -639 | 4 | 6 | 5 | 1212 | 1107 | 4 | 0 | 6 | 1025 | 936 |
| -1 | 7 | 4 | 377 | 364 | -2 | 12 | 4 | 473 | 485 | -6 | 2 | 5 | 1470 | 1409 | 8 | 6 | 5 | 527 | -514 | -7 | 1 | 6 | 471 | 479 |
| 0 | 7 | 4 | 2126 | -2211 | 1 | 12 | 4 | 385 | -393 | -4 | 2 | 5 | 368 | -352 | -7 | 7 | 5 | 856 | 863 | -6 | 1 | 6 | 1014 | 940 |
| 2 | 7 | 4 | 517 | -485 | -5 | 13 | 4 | 337 | 331 | -2 | 2 | 5 | 1386 | -1371 | -5 | 7 | 5 | 343 | 334 | -5 | 1 | 6 | 337 | 296 |
| 4 | 7 | 4 | 1431 | 1335 | -3 | 13 | 4 | 657 | -640 | 0 | 2 | 5 | 1064 | 1108 | -3 | 7 | 5 | 1698 | -1668 | -3 | 1 | 6 | 409 | -401 |
| -7 | 8 | 4 | 398 | 402 | -2 | 13 | 4 | 371 | 375 | 4 | 2 | 5 | 1566 | -1472 | -1 | 7 | 5 | 639 | -666 | -2 | 1 | 6 | 286 | -295 |
| -6 | 8 | 4 | 915 | 885 | -1 | 13 | 4 | 609 | -653 | 6 | 2 | 5 | 573 | 551 | 1 | 7 | 5 | 991 | 961 | -1 | 1 | 6 | 409 | -432 |
| -3 | 8 | 4 | 710 | -688 | 1 | 13 | 4 | 490 | 510 | 8 | 2 | 5 | 737 | 695 | 7 | 7 | 5 | 685 | 646 | 0 | 1 | 6 | 766 | 837 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 | 1 | 6 | 616 | -563 | -4 | 7 | 6 | 308 | -305 | -4 | 2 | 7 | 317 | -299 | 5 | 8 | 7 | 542 | -502 | -4 | 4 | 8 | 470 | -481 |
| 4 | 1 | 6 | 1038 | -958 | -2 | 7 | 6 | 328 | 339 | -2 | 2 | 7 | 983 | -1045 | -3 | 9 | 7 | 373 | 365 | -2 | 4 | 8 | 893 | -905 |
| 7 | 1 | 6 | 673 | 617 | 0 | 7 | 6 | 715 | -747 | 0 | 2 | 7 | 652 | 680 | 0 | 9 | 7 | 933 | -962 | 0 | 4 | 8 | 735 | 782 |
| -7 | 2 | 6 | 491 | -469 | 4 | 7 | 6 | 1210 | 1138 | 2 | 2 | 7 | 872 | 863 | 1 | 9 | 7 | 353 | -357 | 2 | 4 | 8 | 576 | 609 |
| -5 | 2 | 6 | 380 | -345 | 1 | 8 | 6 | 403 | 383 | 4 | 2 | 7 | 442 | -432 | 4 | 9 | 7 | 831 | 764 | 4 | 4 | 8 | 692 | -625 |
| -3 | 2 | 6 | 1171 | 1161 | 4 | 8 | 6 | 670 | 620 | -3 | 3 | 7 | 1055 | 1103 | -6 | 10 | 7 | 504 | 487 | -7 | 5 | 8 | 428 | -424 |
| -1 | 2 | 6 | 362 | -384 | -7 | 9 | 6 | 521 | 502 | 0 | 3 | 7 | 815 | 892 | -2 | 10 | 7 | 700 | -711 | -6 | 5 | 8 | 436 | -393 |
| 1 | 2 | 6 | 1499 | -1625 | -6 | 9 | 6 | 334 | 308 | 1 | 3 | 7 | 1298 | -1399 | 0 | 10 | 7 | 467 | 454 | -3 | 5 | 8 | 1218 | 1204 |
| 3 | 2 | 6 | 837 | 774 | -3 | 9 | 6 | 415 | -403 | 4 | 3 | 7 | 899 | -911 | 2 | 10 | 7 | 522 | 507 | -2 | 5 | 8 | 496 | 471 |
| 5 | 2 | 6 | 893 | 872 | 0 | 9 | 6 | 530 | 537 | 5 | 3 | 7 | 489 | 451 | -3 | 11 | 7 | 744 | 757 | 1 | 5 | 8 | 1002 | -1058 |
| -6 | 3 | 6 | 966 | 869 | 3 | 9 | 6 | 605 | -567 | -7 | 4 | 7 | 331 | -287 | 1 | 11 | 7 | 893 | -892 | -5 | 6 | 8 | 576 | 587 |
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| 0 | 3 | 6 | 809 | 904 | -5 | 10 | 6 | 354 | -336 | -1 | 4 | 7 | 508 | -530 | 1 | 12 | 7 | 499 | -509 | -1 | 6 | 8 | 625 | -632 |
| 3 | 3 | 6 | 427 | 379 | -3 | 10 | 6 | 616 | 596 | 1 | 4 | 7 | 892 | -961 | 3 | 12 | 7 | 437 | 415 | -6 | 7 | 8 | 730 | -707 |
| 4 | 3 | 6 | 1158 | -1119 | 1 | 10 | 6 | 728 | -749 | 3 | 4 | 7 | 693 | 668 | 0 | 13 | 7 | 655 | 693 | -3 | 7 | 8 | 576 | -582 |
| 0 | 4 | 6 | 403 | 428 | 3 | 10 | 6 | 592 | 528 | 5 | 4 | 7 | 673 | 639 | -2 | 14 | 7 | 358 | 400 | -2 | 7 | 8 | 866 | 859 |
| 1 | 4 | 6 | 313 | -303 | 5 | 10 | 6 | 467 | 417 | 7 | 4 | 7 | 539 | -516 | -6 | 0 | 8 | 647 | -641 | 0 | 7 | 8 | 454 | 441 |
| 2 | 4 | 6 | 349 | -343 | -6 | 11 | 6 | 670 | 606 | -3 | 5 | 7 | 669 | -646 | -4 | 0 | 8 | 741 | 713 | 1 | 7 | 8 | 508 | 537 |
| 4 | 4 | 6 | 969 | -898 | 0 | 11 | 6 | 502 | 539 | -2 | 5 | 7 | 396 | 402 | -2 | 0 | 8 | 937 | 987 | -6 | 8 | 8 | 382 | -372 |
| -7 | 5 | 6 | 599 | -616 | 4 | 11 | 6 | 778 | -742 | 0 | 5 | 7 | 1128 | 1198 | 0 | 0 | 8 | 1068 | -1140 | -4 | 8 | 8 | 479 | 471 |
| -6 | 5 | 6 | 517 | -502 | 1 | 12 | 6 | 452 | -451 | 1 | 5 | 7 | 707 | 745 | 2 | 0 | 8 | 529 | -578 | -2 | 8 | 8 | 690 | 681 |
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| 0 | 5 | 6 | 648 | -689 | -3 | 14 | 6 | 341 | -363 | -6 | 6 | 7 | 750 | -702 | -7 | 1 | 8 | 365 | 344 | 2 | 8 | 8 | 501 | -484 |
| 3 | 5 | 6 | 704 | 639 | 1 | 14 | 6 | 455 | 510 | -4 | 6 | 7 | 303 | 275 | -6 | 1 | 8 | 783 | 725 | 4 | 8 | 8 | 598 | 609 |
| 4 | 5 | 6 | 697 | 641 | 0 | 15 | 6 | 374 | -425 | -2 | 6 | 7 | 991 | 1005 | -3 | 1 | 8 | 1165 | -1130 | -3 | 9 | 8 | 913 | -909 |
| 7 | 5 | 6 | 662 | -612 | -7 | 0 | 7 | 555 | 521 | 0 | 6 | 7 | 716 | -696 | -2 | 1 | 8 | 941 | -991 | 1 | 9 | 8 | 834 | 830 |
| -9 | 6 | 6 | 400 | -413 | -3 | 0 | 7 | 845 | -809 | 2 | 6 | 7 | 746 | -749 | 1 | 1 | 8 | 1017 | 1085 | -5 | 10 | 8 | 424 | -427 |
| -7 | 6 | 6 | 440 | 454 | -1 | 0 | 7 | 694 | 782 | 4 | 6 | 7 | 468 | 419 | -5 | 2 | 8 | 575 | -551 | -3 | 10 | 8 | 396 | 375 |
| -5 | 6 | 6 | 412 | 397 | 1 | 0 | 7 | 1069 | 1112 | -3 | 7 | 7 | 1030 | -1034 | -3 | 2 | 8 | 411 | 388 | -1 | 10 | 8 | 450 | 435 |
| -3 | 6 | 6 | 1037 | -1018 | 3 | 0 | 7 | 800 | -777 | 0 | 7 | 7 | 543 | -579 | -1 | 2 | 8 | 635 | 674 | -2 | 11 | 8 | 633 | -613 |
| -1 | 6 | 6 | 344 | 345 | -3 | 1 | 7 | 1168 | 1132 | 1 | 7 | 7 | 1244 | 1267 | -6 | 3 | 8 | 776 | 733 | -2 | 12 | 8 | 426 | -423 |
| 0 | 6 | 6 | 312 | 329 | 0 | 1 | 7 | 1090 | -1151 | 4 | 7 | 7 | 554 | 538 | -3 | 3 | 8 | 827 | 861 | 0 | 12 | 8 | 472 | 477 |
| 1 | 6 | 6 | 1276 | 1292 | 1 | 1 | 7 | 1257 | -1320 | -3 | 8 | 7 | 536 | -544 | -2 | 3 | 8 | 773 | -808 | -5 | 0 | 9 | 573 | 544 |
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| 5 | 6 | 6 | 748 | -685 | 5 | 1 | 7 | 458 | 432 | 1 | 8 | 7 | 852 | 885 | 1 | 3 | 8 | 796 | -862 | 1 | 0 | 9 | 699 | 771 |
| -6 | 7 | 6 | 872 | -859 | -6 | 2 | 7 | 795 | 731 | 3 | 8 | 7 | 655 | -591 | -6 | 4 | 8 | 534 | 511 | 3 | 0 | 9 | 577 | -562 |

OBSERVED AND CALCULATED STRUCTURE FACTORS

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -6 | 1 | 9 | 677 | -656 | -2 | 3 | 9 | 426 | -453 | 4 | 5 | 9 | 617 | -581 | 0 | 0 | 10 | 606 | -696 | 0 | 8 | 10 | 437 | -430 |
| -5 | 1 | 9 | 346 | -335 | -7 | 4 | 9 | 400 | -405 | -4 | 6 | 9 | 389 | -386 | 4 | 0 | 10 | 721 | 711 | -3 | 0 | 11 | 519 | -530 |
| -2 | 1 | 9 | 591 | 619 | -5 | 4 | 9 | 492 | -488 | -2 | 7 | 9 | 348 | 341 | 1 | 1 | 10 | 680 | 707 | -2 | 1 | 11 | 595 | 601 |
| 4 | 1 | 9 | 578 | 529 | -3 | 4 | 9 | 754 | 755 | -5 | 8 | 9 | 436 | 388 | 1 | 3 | 10 | 493 | -547 | 1 | 1 | 11 | 437 | 470 |
| -6 | 2 | 9 | 346 | 317 | -1 | 4 | 9 | 343 | 348 | -3 | 8 | 9 | 534 | -520 | -2 | 4 | 10 | 354 | -378 | 0 | 2 | 11 | 505 | -534 |
| -4 | 2 | 9 | 423 | 410 | 1 | 4 | 9 | 568 | -599 | 1 | 8 | 9 | 425 | 455 | 0 | 4 | 10 | 537 | 603 | -2 | 3 | 11 | 511 | -512 |
| -6 | 3 | 9 | 514 | 519 | -6 | 5 | 9 | 715 | 680 | -2 | 9 | 9 | 478 | 462 | 1 | 5 | 10 | 689 | -750 | 1 | 3 | 11 | 406 | 427 |
| -5 | 3 | 9 | 364 | -357 | -2 | 5 | 9 | 617 | -656 | -6 | 0 | 10 | 500 | -483 | | | | | | | | | | |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 4 | 0 | 0 | 1947 | -2132 | 10 | 2 | 0 | 334 | 380 | 7 | 4 | 0 | 626 | 625 | 1 | 6 | 0 | 1516 | -1474 | 2 | 8 | 0 | 155 | 111 |
| 6 | 0 | 0 | 227 | 242 | 11 | 2 | 0 | 679 | -739 | 8 | 4 | 0 | 458 | 471 | 2 | 6 | 0 | 335 | -355 | 5 | 8 | 0 | 488 | -498 |
| 8 | 0 | 0 | 893 | -960 | 12 | 2 | 0 | 1276 | -1417 | 10 | 4 | 0 | 331 | 342 | 3 | 6 | 0 | 321 | -327 | 6 | 8 | 0 | 121 | -77 |
| 10 | 0 | 0 | 1176 | -1210 | 13 | 2 | 0 | 155 | 255 | 11 | 4 | 0 | 120 | -118 | 4 | 6 | 0 | 505 | -522 | 7 | 8 | 0 | 555 | 583 |
| 12 | 0 | 0 | 2290 | 2518 | 14 | 2 | 0 | 794 | 850 | 12 | 4 | 0 | 507 | -517 | 5 | 6 | 0 | 994 | 1005 | 8 | 8 | 0 | 120 | 125 |
| 14 | 0 | 0 | 1306 | -1405 | 15 | 2 | 0 | 332 | 372 | 13 | 4 | 0 | 326 | 321 | 6 | 6 | 0 | 559 | 586 | 10 | 8 | 0 | 129 | -66 |
| 16 | 0 | 0 | 838 | -874 | 16 | 2 | 0 | 223 | 263 | 14 | 4 | 0 | 161 | -123 | 7 | 6 | 0 | 949 | -958 | 11 | 8 | 0 | 573 | -590 |
| 18 | 0 | 0 | 1229 | 1273 | 17 | 2 | 0 | 306 | -347 | 15 | 4 | 0 | 268 | -280 | 8 | 6 | 0 | 350 | -354 | 12 | 8 | 0 | 131 | -69 |
| 20 | 0 | 0 | 535 | -557 | 18 | 2 | 0 | 743 | -781 | 16 | 4 | 0 | 495 | 490 | 9 | 6 | 0 | 187 | 180 | 13 | 8 | 0 | 503 | 505 |
| 22 | 0 | 0 | 233 | -248 | 20 | 2 | 0 | 225 | 249 | 17 | 4 | 0 | 169 | -165 | 10 | 6 | 0 | 248 | -260 | 1 | 9 | 0 | 159 | 176 |
| 24 | 0 | 0 | 615 | 593 | 23 | 2 | 0 | 216 | -214 | 18 | 4 | 0 | 238 | -246 | 11 | 6 | 0 | 858 | 871 | 2 | 9 | 0 | 146 | -107 |
| 1 | 1 | 0 | 552 | -524 | 24 | 2 | 0 | 161 | -189 | 19 | 4 | 0 | 202 | 201 | 12 | 6 | 0 | 560 | 608 | 3 | 9 | 0 | 342 | -350 |
| 2 | 1 | 0 | 3326 | -3458 | 3 | 3 | 0 | 323 | -321 | 22 | 4 | 0 | 178 | 187 | 13 | 6 | 0 | 765 | -774 | 4 | 9 | 0 | 130 | 46 |
| 3 | 1 | 0 | 526 | 529 | 4 | 3 | 0 | 522 | -524 | 1 | 5 | 0 | 629 | 584 | 14 | 6 | 0 | 188 | -187 | 5 | 9 | 0 | 205 | 176 |
| 4 | 1 | 0 | 1447 | 1509 | 6 | 3 | 0 | 265 | 258 | 2 | 5 | 0 | 1055 | 1051 | 16 | 6 | 0 | 322 | -329 | 9 | 9 | 0 | 184 | -187 |
| 6 | 1 | 0 | 375 | -380 | 7 | 3 | 0 | 183 | 171 | 3 | 5 | 0 | 1360 | -1286 | 17 | 6 | 0 | 549 | 543 | -25 | 0 | 1 | 229 | -223 |
| 8 | 1 | 0 | 939 | -1026 | 8 | 3 | 0 | 131 | 132 | 4 | 5 | 0 | 763 | -744 | 18 | 6 | 0 | 394 | 401 | -23 | 0 | 1 | 274 | 289 |
| 9 | 1 | 0 | 337 | 382 | 9 | 3 | 0 | 128 | -116 | 5 | 5 | 0 | 429 | 411 | 19 | 6 | 0 | 440 | -451 | -19 | 0 | 1 | 203 | -201 |
| 10 | 1 | 0 | 1599 | 1760 | 10 | 3 | 0 | 206 | -219 | 6 | 5 | 0 | 237 | -238 | 1 | 7 | 0 | 872 | -890 | -17 | 0 | 1 | 307 | 326 |
| 12 | 1 | 0 | 233 | 249 | 11 | 3 | 0 | 244 | -244 | 7 | 5 | 0 | 553 | 542 | 2 | 7 | 0 | 372 | -377 | -15 | 0 | 1 | 161 | -151 |
| 13 | 1 | 0 | 258 | -482 | 12 | 3 | 0 | 112 | 106 | 8 | 5 | 0 | 614 | 628 | 3 | 7 | 0 | 1043 | 1050 | -13 | 0 | 1 | 391 | -416 |
| 14 | 1 | 0 | 1756 | -1896 | 13 | 3 | 0 | 461 | 472 | 9 | 5 | 0 | 683 | -685 | 4 | 7 | 0 | 282 | 277 | -11 | 0 | 1 | 574 | 636 |
| 16 | 1 | 0 | 1371 | 1446 | 15 | 3 | 0 | 233 | -253 | 10 | 5 | 0 | 410 | -430 | 5 | 7 | 0 | 552 | -550 | 5 | 0 | 1 | 142 | 155 |
| 18 | 1 | 0 | 156 | -160 | 16 | 3 | 0 | 419 | -447 | 11 | 5 | 0 | 346 | 328 | 6 | 7 | 0 | 140 | -98 | 7 | 0 | 1 | 137 | -132 |
| 20 | 1 | 0 | 522 | -550 | 18 | 3 | 0 | 225 | 227 | 12 | 5 | 0 | 249 | -234 | 7 | 7 | 0 | 207 | -230 | 11 | 0 | 1 | 103 | 132 |
| 22 | 1 | 0 | 303 | 304 | 19 | 3 | 0 | 139 | 150 | 13 | 5 | 0 | 219 | 214 | 8 | 7 | 0 | 206 | -195 | 15 | 0 | 1 | 258 | 262 |
| 25 | 1 | 0 | 139 | -172 | 20 | 3 | 0 | 125 | -141 | 14 | 5 | 0 | 570 | 599 | 9 | 7 | 0 | 960 | 1000 | 17 | 0 | 1 | 377 | 415 |
| 0 | 2 | 0 | 4044 | -3959 | 23 | 3 | 0 | 123 | -148 | 15 | 5 | 0 | 478 | -463 | 10 | 7 | 0 | 275 | 295 | 19 | 0 | 1 | 384 | -391 |
| 1 | 2 | 0 | 1329 | 1226 | 24 | 3 | 0 | 118 | 147 | 16 | 5 | 0 | 294 | -317 | 11 | 7 | 0 | 334 | -325 | 23 | 0 | 1 | 168 | 161 |
| 2 | 2 | 0 | 2408 | 2321 | 0 | 4 | 0 | 713 | -693 | 17 | 5 | 0 | 390 | 390 | 13 | 7 | 0 | 497 | -526 | 25 | 0 | 1 | 241 | -205 |
| 3 | 2 | 0 | 187 | -157 | 1 | 4 | 0 | 353 | 335 | 18 | 5 | 0 | 159 | -157 | 14 | 7 | 0 | 258 | -272 | -25 | 1 | 1 | 247 | 235 |
| 4 | 2 | 0 | 309 | 333 | 2 | 4 | 0 | 139 | -116 | 19 | 5 | 0 | 193 | 212 | 15 | 7 | 0 | 715 | 732 | -24 | 1 | 1 | 193 | 216 |
| 5 | 2 | 0 | 254 | -248 | 3 | 4 | 0 | 358 | 349 | 20 | 5 | 0 | 350 | 384 | 16 | 7 | 0 | 231 | 227 | -22 | 1 | 1 | 267 | -284 |
| 6 | 2 | 0 | 388 | -389 | 4 | 4 | 0 | 644 | 640 | 21 | 5 | 0 | 381 | -378 | 17 | 7 | 0 | 238 | -241 | -21 | 1 | 1 | 204 | -231 |
| 8 | 2 | 0 | 318 | 312 | 5 | 4 | 0 | 375 | -366 | 22 | 5 | 0 | 161 | -182 | 0 | 8 | 0 | 229 | -80 | -19 | 1 | 1 | 182 | 197 |
| 9 | 2 | 0 | 293 | 315 | 6 | 4 | 0 | 590 | -591 | 0 | 6 | 0 | 1031 | 1086 | 1 | 8 | 0 | 800 | 834 | -18 | 1 | 1 | 306 | 292 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -16 | 1 | 1 | 567 | -600 | -14 | 2 | 1 | 1407 | 1482 | -8 | 3 | 1 | 149 | 169 | -5 | 4 | 1 | 282 | 275 | -10 | 5 | 1 | 590 | -647 |
| -15 | 1 | 1 | 273 | -307 | -13 | 2 | 1 | 132 | -235 | -7 | 3 | 1 | 775 | -799 | -4 | 4 | 1 | 703 | 691 | -9 | 5 | 1 | 552 | 547 |
| -14 | 1 | 1 | 140 | 166 | -9 | 2 | 1 | 786 | 846 | -6 | 3 | 1 | 1701 | -1683 | -3 | 4 | 1 | 862 | -838 | -8 | 5 | 1 | 156 | -131 |
| -13 | 1 | 1 | 530 | 594 | -8 | 2 | 1 | 1515 | 1588 | -5 | 3 | 1 | 836 | 831 | -2 | 4 | 1 | 1372 | -1371 | -7 | 5 | 1 | 289 | 296 |
| -12 | 1 | 1 | 1602 | 1655 | -7 | 2 | 1 | 143 | -166 | -4 | 3 | 1 | 965 | 968 | -1 | 4 | 1 | 1186 | 1118 | -6 | 5 | 1 | 389 | 400 |
| -10 | 1 | 1 | 942 | -976 | -6 | 2 | 1 | 154 | -161 | 0 | 3 | 1 | 2778 | -2721 | 0 | 4 | 1 | 645 | 665 | -5 | 5 | 1 | 461 | -467 |
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 8 | 6 | 3 | 354 | 345 | 4 | 8 | 3 | 200 | 178 | 10 | 0 | 4 | 1632 | 1673 | 14 | 1 | 4 | 323 | 330 | 10 | 2 | 4 | 1077 | -1100 |
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| 1 | 7 | 3 | 241 | -239 | -2 | 9 | 3 | 118 | 5 | -16 | 1 | 4 | 176 | 174 | -16 | 2 | 4 | 408 | 420 | -20 | 3 | 4 | 250 | -224 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 21 | 3 | 4 | 267 | -265 | -19 | 5 | 4 | 293 | 265 | 19 | 5 | 4 | 264 | -238 | -17 | 7 | 4 | 356 | 331 | 3 | 8 | 4 | 690 | -715 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 11 | 3 | 6 | 334 | 321 | 11 | 4 | 6 | 241 | 207 | 13 | 5 | 6 | 529 | -485 | 16 | 6 | 6 | 220 | 207 | 9 | 8 | 6 | 485 | -478 |
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| 13 | 3 | 6 | 101 | -87 | 13 | 4 | 6 | 124 | 164 | 16 | 5 | 6 | 217 | -198 | -16 | 7 | 6 | 134 | 78 | 11 | 8 | 6 | 428 | 414 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 13 | 1 | 7 | 288 | 282 | 13 | 2 | 7 | 301 | 305 | 15 | 3 | 7 | 401 | -370 | 15 | 4 | 7 | 366 | 352 | -15 | 6 | 7 | 338 | -301 |
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| 16 | 1 | 7 | 404 | 375 | 15 | 2 | 7 | 280 | -284 | 17 | 3 | 7 | 390 | 361 | 18 | 4 | 7 | 335 | 308 | -11 | 6 | 7 | 191 | 168 |
| 17 | 1 | 7 | 178 | -169 | 16 | 2 | 7 | 138 | -126 | 18 | 3 | 7 | 285 | 263 | 19 | 4 | 7 | 322 | -317 | -8 | 6 | 7 | 218 | 216 |
| 18 | 1 | 7 | 125 | -132 | 18 | 2 | 7 | 577 | -532 | 20 | 3 | 7 | 263 | 240 | 20 | 4 | 7 | 326 | -304 | -6 | 6 | 7 | 164 | -166 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 1 | 7 | 7 | 223 | -227 | 22 | 0 | 8 | 492 | 420 | -13 | 2 | 8 | 360 | 334 | 2 | 3 | 8 | 593 | -530 | -11 | 5 | 8 | 502 | -453 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 7 | 6 | 8 | 302 | 303 | 1 | 8 | 8 | 219 | -192 | -16 | 2 | 9 | 530 | 456 | -14 | 3 | 9 | 665 | -599 | -12 | 4 | 9 | 233 | 211 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 8 | 7 | 10 | 115 | 135 | 18 | 1 | 11 | 185 | -206 | -13 | 3 | 11 | 108 | 133 | -5 | 4 | 11 | 106 | -127 | 8 | 5 | 11 | 45 | 205 |
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| 7 | 1 | 11 | 69 | 91 | 16 | 2 | 11 | 349 | -373 | -16 | 4 | 11 | 116 | -137 | -5 | 5 | 11 | 260 | 280 | 12 | 0 | 12 | 377 | -405 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
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| 10 | 1 | 12 | 398 | -426 | -11 | 4 | 12 | 134 | -137 | -6 | 6 | 12 | 138 | -154 | 14 | 1 | 13 | 114 | 109 | -1 | 3 | 13 | 89 | 76 |
| 12 | 1 | 12 | 119 | 130 | -10 | 4 | 12 | 87 | -138 | -5 | 6 | 12 | 258 | 263 | -18 | 2 | 13 | 80 | 90 | 0 | 3 | 13 | 277 | 297 |
| 14 | 1 | 12 | 129 | 122 | -7 | 4 | 12 | 116 | 140 | -4 | 6 | 12 | 75 | 76 | -14 | 2 | 13 | 88 | -88 | 1 | 3 | 13 | 174 | -176 |
| 16 | 1 | 12 | 335 | -353 | 0 | 4 | 12 | 128 | 158 | -1 | 6 | 12 | 164 | -187 | -12 | 2 | 13 | 137 | 153 | 2 | 3 | 13 | 211 | -225 |
| 18 | 1 | 12 | 183 | 187 | 1 | 4 | 12 | 184 | -210 | 0 | 6 | 12 | 126 | -140 | -9 | 2 | 13 | 131 | -122 | 3 | 3 | 13 | 75 | 93 |
| -18 | 2 | 12 | 126 | 127 | 2 | 4 | 12 | 235 | -283 | 1 | 6 | 12 | 321 | 340 | -8 | 2 | 13 | 370 | -393 | 4 | 3 | 13 | 111 | 110 |
| -14 | 2 | 12 | 71 | -75 | 3 | 4 | 12 | 117 | 157 | 2 | 6 | 12 | 192 | 187 | -7 | 2 | 13 | 156 | 164 | 6 | 3 | 13 | 239 | 265 |
| -12 | 2 | 12 | 138 | 165 | 5 | 4 | 12 | 108 | 152 | 3 | 6 | 12 | 232 | -257 | -6 | 2 | 13 | 383 | 403 | 7 | 3 | 13 | 270 | -288 |
| -10 | 2 | 12 | 199 | -213 | 6 | 4 | 12 | 176 | 200 | 5 | 6 | 12 | 177 | -192 | -4 | 2 | 13 | 89 | 109 | 8 | 3 | 13 | 591 | -630 |
| -9 | 2 | 12 | 125 | 152 | 10 | 4 | 12 | 83 | -96 | 6 | 6 | 12 | 205 | -225 | -3 | 2 | 13 | 129 | -140 | 9 | 3 | 13 | 251 | 240 |
| -6 | 2 | 12 | 327 | 349 | 12 | 4 | 12 | 110 | 138 | 7 | 6 | 12 | 321 | 342 | -2 | 2 | 13 | 269 | -267 | 10 | 3 | 13 | 111 | 104 |
| -5 | 2 | 12 | 148 | -149 | 13 | 4 | 12 | 99 | -87 | 8 | 6 | 12 | 114 | 138 | -1 | 2 | 13 | 107 | 103 | 12 | 3 | 13 | 229 | 240 |
| -4 | 2 | 12 | 332 | -346 | 14 | 4 | 12 | 68 | -63 | 9 | 6 | 12 | 92 | -87 | 0 | 2 | 13 | 164 | 164 | 13 | 3 | 13 | 137 | -153 |
| -3 | 2 | 12 | 105 | 113 | -15 | 5 | 12 | 141 | 142 | -3 | 7 | 12 | 227 | -236 | 2 | 2 | 13 | 134 | 146 | 14 | 3 | 13 | 236 | -227 |
| 0 | 2 | 12 | 112 | 146 | -14 | 5 | 12 | 148 | 142 | -2 | 7 | 12 | 85 | -78 | 3 | 2 | 13 | 90 | -90 | 15 | 3 | 13 | 125 | 109 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -14 | 4 | 13 | 75 | 78 | 9 | 5 | 13 | 250 | -240 | -7 | 2 | 14 | 91 | -82 | -2 | 6 | 14 | 79 | -56 | -2 | 3 | 15 | 217 | 190 |
| -12 | 4 | 13 | 80 | -65 | 10 | 5 | 13 | 101 | -106 | -6 | 2 | 14 | 102 | 134 | 1 | 6 | 14 | 153 | 148 | 0 | 3 | 15 | 86 | 99 |
| -10 | 4 | 13 | 74 | -79 | -7 | 6 | 13 | 135 | 128 | -5 | 2 | 14 | 75 | -65 | 2 | 6 | 14 | 133 | 102 | 2 | 3 | 15 | 109 | -79 |
| -9 | 4 | 13 | 128 | 135 | -5 | 6 | 13 | 147 | -146 | -4 | 2 | 14 | 402 | -378 | 3 | 6 | 14 | 180 | -206 | 3 | 3 | 15 | 93 | 91 |
| -8 | 4 | 13 | 282 | 301 | 5 | 6 | 13 | 129 | 109 | -3 | 2 | 14 | 162 | 150 | -15 | 0 | 15 | 127 | 89 | 4 | 3 | 15 | 205 | 184 |
| -7 | 4 | 13 | 293 | -300 | 6 | 6 | 13 | 96 | 80 | -2 | 2 | 14 | 240 | 224 | -14 | 1 | 15 | 91 | -74 | 7 | 3 | 15 | 84 | -77 |
| -6 | 4 | 13 | 284 | -273 | 7 | 6 | 13 | 94 | -104 | 1 | 2 | 14 | 73 | -46 | -10 | 1 | 15 | 76 | 26 | 8 | 3 | 15 | 260 | -253 |
| -5 | 4 | 13 | 173 | 170 | -16 | 0 | 14 | 344 | 341 | 2 | 2 | 14 | 197 | -200 | -8 | 1 | 15 | 100 | -107 | 9 | 3 | 15 | 151 | 140 |
| -3 | 4 | 13 | 120 | 98 | -14 | 0 | 14 | 153 | -153 | 8 | 2 | 14 | 187 | -180 | -4 | 1 | 15 | 182 | 181 | 10 | 3 | 15 | 233 | 193 |
| -2 | 4 | 13 | 236 | 231 | -10 | 0 | 14 | 169 | 176 | 9 | 2 | 14 | 121 | 94 | -2 | 1 | 15 | 120 | -108 | 11 | 3 | 15 | 90 | -67 |
| -1 | 4 | 13 | 259 | -242 | -8 | 0 | 14 | 138 | -149 | 10 | 2 | 14 | 219 | 199 | 2 | 1 | 15 | 124 | 111 | -7 | 4 | 15 | 85 | -70 |
| 3 | 4 | 13 | 148 | 152 | -6 | 0 | 14 | 303 | -311 | 14 | 2 | 14 | 141 | -131 | 4 | 1 | 15 | 73 | -69 | -6 | 4 | 15 | 202 | -177 |
| 4 | 4 | 13 | 225 | 240 | -4 | 0 | 14 | 545 | 551 | -15 | 3 | 14 | 75 | -57 | 8 | 1 | 15 | 144 | 144 | -5 | 4 | 15 | 207 | 187 |
| 5 | 4 | 13 | 242 | -222 | -2 | 0 | 14 | 400 | -388 | -5 | 3 | 14 | 116 | 103 | 10 | 1 | 15 | 109 | -89 | -4 | 4 | 15 | 140 | 123 |
| 6 | 4 | 13 | 325 | -308 | 0 | 0 | 14 | 109 | -116 | -2 | 3 | 14 | 77 | 55 | -14 | 2 | 15 | 90 | -97 | -2 | 4 | 15 | 173 | 176 |
| 7 | 4 | 13 | 165 | 145 | 2 | 0 | 14 | 339 | 325 | 0 | 3 | 14 | 93 | -105 | -13 | 2 | 15 | 83 | 71 | -1 | 4 | 15 | 250 | -223 |
| 8 | 4 | 13 | 73 | -52 | 4 | 0 | 14 | 226 | -227 | 12 | 3 | 14 | 83 | -80 | -12 | 2 | 15 | 104 | 98 | 0 | 4 | 15 | 228 | -221 |
| 9 | 4 | 13 | 196 | 194 | 6 | 0 | 14 | 91 | -111 | -8 | 4 | 14 | 90 | 118 | -8 | 2 | 15 | 97 | -105 | 1 | 4 | 15 | 137 | 112 |
| 10 | 4 | 13 | 318 | 317 | 8 | 0 | 14 | 420 | 419 | 1 | 4 | 14 | 81 | -110 | -7 | 2 | 15 | 81 | 68 | 2 | 4 | 15 | 99 | 49 |
| 11 | 4 | 13 | 233 | -224 | 10 | 0 | 14 | 319 | -287 | 2 | 4 | 14 | 140 | -145 | -6 | 2 | 15 | 265 | 255 | 4 | 4 | 15 | 120 | 127 |
| 12 | 4 | 13 | 182 | -180 | 12 | 0 | 14 | 146 | -119 | 3 | 4 | 14 | 75 | 86 | -5 | 2 | 15 | 86 | -66 | 5 | 4 | 15 | 134 | -131 |
| -12 | 5 | 13 | 96 | -91 | 14 | 0 | 14 | 361 | 337 | 6 | 4 | 14 | 100 | 96 | -4 | 2 | 15 | 137 | -130 | 6 | 4 | 15 | 181 | -156 |
| -11 | 5 | 13 | 74 | 62 | -8 | 1 | 14 | 216 | -221 | 8 | 4 | 14 | 106 | -106 | -2 | 2 | 15 | 233 | -230 | 7 | 4 | 15 | 136 | 127 |
| -10 | 5 | 13 | 74 | 55 | -7 | 1 | 14 | 73 | 57 | -8 | 5 | 14 | 102 | 100 | -1 | 2 | 15 | 124 | 115 | 8 | 4 | 15 | 98 | 81 |
| -9 | 5 | 13 | 107 | -115 | -6 | 1 | 14 | 359 | 344 | -7 | 5 | 14 | 160 | -172 | 0 | 2 | 15 | 286 | 275 | -7 | 5 | 15 | 86 | 53 |
| -6 | 5 | 13 | 141 | -138 | -4 | 1 | 14 | 82 | -57 | -6 | 5 | 14 | 97 | -126 | 1 | 2 | 15 | 88 | -58 | -4 | 5 | 15 | 82 | 87 |
| -5 | 5 | 13 | 269 | 265 | -2 | 1 | 14 | 370 | -369 | -3 | 5 | 14 | 151 | 161 | 6 | 2 | 15 | 237 | 231 | -3 | 5 | 15 | 187 | -174 |
| -4 | 5 | 13 | 251 | 249 | 0 | 1 | 14 | 323 | 320 | -2 | 5 | 14 | 91 | 118 | 8 | 2 | 15 | 140 | -129 | -2 | 5 | 15 | 138 | -117 |
| -3 | 5 | 13 | 263 | -264 | 4 | 1 | 14 | 262 | -261 | -1 | 5 | 14 | 138 | -125 | 10 | 2 | 15 | 184 | -184 | -1 | 5 | 15 | 134 | 123 |
| -2 | 5 | 13 | 96 | -93 | 6 | 1 | 14 | 309 | 315 | 4 | 5 | 14 | 89 | 88 | 12 | 2 | 15 | 244 | 211 | 2 | 5 | 15 | 123 | 87 |
| 0 | 5 | 13 | 87 | -103 | 10 | 1 | 14 | 377 | -359 | 5 | 5 | 14 | 188 | -179 | -10 | 3 | 15 | 117 | -115 | 3 | 5 | 15 | 131 | -71 |
| 1 | 5 | 13 | 120 | 151 | 12 | 1 | 14 | 335 | 303 | 6 | 5 | 14 | 177 | -166 | -9 | 3 | 15 | 78 | 62 | -10 | 0 | 16 | 115 | 109 |
| 2 | 5 | 13 | 99 | 111 | -16 | 2 | 14 | 187 | -154 | 7 | 5 | 14 | 128 | 114 | -8 | 3 | 15 | 120 | 104 | -4 | 0 | 16 | 153 | 139 |
| 6 | 5 | 13 | 122 | -109 | -15 | 2 | 14 | 86 | 87 | -5 | 6 | 14 | 195 | 180 | -5 | 3 | 15 | 111 | -83 | -2 | 0 | 16 | 391 | -341 |
| 7 | 5 | 13 | 258 | 244 | -14 | 2 | 14 | 97 | 101 | -4 | 6 | 14 | 133 | 120 | -4 | 3 | 15 | 291 | -265 | 0 | 0 | 16 | 133 | 68 |
| 8 | 5 | 13 | 254 | 228 | -10 | 2 | 14 | 84 | -25 | -3 | 6 | 14 | 200 | -183 | -3 | 3 | 15 | 218 | 192 | 2 | 0 | 16 | 269 | 228 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 4 | 0 | 16 | 195 | -151 | 2 | 1 | 16 | 281 | -234 | 8 | 2 | 16 | 98 | -85 | 0 | 2 | 17 | 154 | 131 | 3 | 3 | 17 | 102 | 99 |
| 8 | 0 | 16 | 164 | 158 | 6 | 1 | 16 | 132 | 114 | 10 | 2 | 16 | 86 | 78 | 1 | 2 | 17 | 87 | -55 | 4 | 3 | 17 | 230 | 189 |
| 10 | 0 | 16 | 272 | -228 | 8 | 1 | 16 | 127 | -93 | 1 | 3 | 16 | 85 | 60 | 2 | 2 | 17 | 152 | -113 | -6 | 0 | 18 | 91 | 71 |
| -12 | 1 | 16 | 194 | 160 | -4 | 2 | 16 | 96 | -70 | -2 | 4 | 16 | 101 | 80 | 6 | 2 | 17 | 130 | 98 | -2 | 0 | 18 | 149 | -122 |
| -6 | 1 | 16 | 146 | 133 | -2 | 2 | 16 | 225 | 193 | -8 | 1 | 17 | 82 | -63 | -4 | 3 | 17 | 102 | -108 | 0 | 0 | 18 | 177 | 128 |
| -4 | 1 | 16 | 161 | -140 | -1 | 2 | 16 | 111 | -77 | -2 | 1 | 17 | 108 | -99 | -2 | 3 | 17 | 133 | 115 | 0 | 1 | 18 | 112 | 100 |
| -2 | 1 | 16 | 77 | -84 | 2 | 2 | 16 | 175 | -154 | 4 | 1 | 17 | 110 | -91 | 0 | 3 | 17 | 114 | -82 | 2 | 1 | 18 | 179 | -134 |
| 0 | 1 | 16 | 366 | 310 | 4 | 2 | 16 | 86 | 61 | -4 | 2 | 17 | 92 | -83 | 2 | 3 | 17 | 90 | -82 | | | | | |

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR 2-Dichloro(butyl)telluro-N-dimethylbenzylammonium Chloride 

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2 | 0 | 0 | 430 | 438 | 13 | 2 | 0 | 184 | -171 | 12 | 5 | 0 | 213 | 213 | -9 | 0 | 1 | 1216 | -1224 | 10 | 1 | 1 | 389 | -385 |
| 4 | 0 | 0 | 2918 | -2945 | 14 | 2 | 0 | 147 | -152 | 14 | 5 | 0 | 256 | -246 | -7 | 0 | 1 | 382 | 359 | 11 | 1 | 1 | 426 | 415 |
| 6 | 0 | 0 | 1860 | -1853 | 15 | 2 | 0 | 403 | -404 | 0 | 6 | 0 | 1240 | 1222 | -5 | 0 | 1 | 2487 | 2467 | 12 | 1 | 1 | 285 | 285 |
| 8 | 0 | 0 | 418 | -428 | 1 | 3 | 0 | 1776 | 1723 | 1 | 6 | 0 | 301 | 290 | -3 | 0 | 1 | 1792 | 1756 | 13 | 1 | 1 | 229 | 236 |
| 10 | 0 | 0 | 485 | 495 | 2 | 3 | 0 | 509 | 484 | 2 | 6 | 0 | 131 | 143 | 1 | 0 | 1 | 1930 | -2115 | 14 | 1 | 1 | 476 | 467 |
| 12 | 0 | 0 | 905 | 908 | 3 | 3 | 0 | 252 | -254 | 3 | 6 | 0 | 398 | 401 | 3 | 0 | 1 | 2011 | -2060 | 16 | 1 | 1 | 138 | 126 |
| 14 | 0 | 0 | 460 | 454 | 4 | 3 | 0 | 554 | 538 | 4 | 6 | 0 | 364 | -363 | 5 | 0 | 1 | 533 | -506 | -16 | 2 | 1 | 268 | -268 |
| 16 | 0 | 0 | 126 | -143 | 5 | 3 | 0 | 1375 | -1387 | 5 | 6 | 0 | 235 | 235 | 7 | 0 | 1 | 2084 | 2097 | -15 | 2 | 1 | 113 | -133 |
| 1 | 1 | 0 | 1060 | -1024 | 6 | 3 | 0 | 98 | -124 | 6 | 6 | 0 | 517 | -526 | 9 | 0 | 1 | 1457 | 1457 | -14 | 2 | 1 | 549 | -560 |
| 2 | 1 | 0 | 1357 | 1369 | 7 | 3 | 0 | 892 | -878 | 7 | 6 | 0 | 167 | -162 | 11 | 0 | 1 | 315 | -306 | -13 | 2 | 1 | 147 | 155 |
| 3 | 1 | 0 | 83 | -94 | 8 | 3 | 0 | 365 | -368 | 8 | 6 | 0 | 298 | -290 | 13 | 0 | 1 | 913 | -910 | -11 | 2 | 1 | 323 | 323 |
| 4 | 1 | 0 | 640 | 652 | 10 | 3 | 0 | 187 | -180 | 9 | 6 | 0 | 330 | -322 | 15 | 0 | 1 | 546 | -535 | -10 | 2 | 1 | 360 | 349 |
| 5 | 1 | 0 | 766 | 735 | 11 | 3 | 0 | 400 | 393 | 12 | 6 | 0 | 398 | 400 | -16 | 1 | 1 | 269 | -256 | -9 | 2 | 1 | 201 | 206 |
| 6 | 1 | 0 | 294 | 303 | 13 | 3 | 0 | 741 | 741 | 1 | 7 | 0 | 857 | -844 | -15 | 1 | 1 | 387 | 384 | -8 | 2 | 1 | 500 | 509 |
| 7 | 1 | 0 | 665 | 659 | 14 | 3 | 0 | 124 | 134 | 2 | 7 | 0 | 323 | 321 | -13 | 1 | 1 | 580 | 586 | -7 | 2 | 1 | 394 | -408 |
| 8 | 1 | 0 | 628 | -629 | 15 | 3 | 0 | 201 | 203 | 4 | 7 | 0 | 153 | 148 | -12 | 1 | 1 | 534 | 537 | -6 | 2 | 1 | 131 | 131 |
| 9 | 1 | 0 | 180 | 200 | 0 | 4 | 0 | 2111 | -2049 | 5 | 7 | 0 | 741 | 725 | -11 | 1 | 1 | 150 | -157 | -5 | 2 | 1 | 488 | -500 |
| 10 | 1 | 0 | 748 | -739 | 1 | 4 | 0 | 755 | 727 | 7 | 7 | 0 | 592 | 593 | -10 | 1 | 1 | 728 | 721 | -4 | 2 | 1 | 264 | 250 |
| 11 | 1 | 0 | 200 | -206 | 2 | 4 | 0 | 518 | -515 | 8 | 7 | 0 | 209 | -181 | -9 | 1 | 1 | 519 | -523 | -3 | 2 | 1 | 334 | 314 |
| 12 | 1 | 0 | 121 | -67 | 3 | 4 | 0 | 698 | 688 | 10 | 7 | 0 | 140 | -126 | -8 | 1 | 1 | 114 | -110 | -2 | 2 | 1 | 238 | 205 |
| 13 | 1 | 0 | 517 | -521 | 4 | 4 | 0 | 772 | 769 | 11 | 7 | 0 | 323 | -310 | -7 | 1 | 1 | 606 | -595 | -1 | 2 | 1 | 281 | 273 |
| 14 | 1 | 0 | 347 | 351 | 5 | 4 | 0 | 445 | 442 | 1 | 8 | 0 | 243 | -248 | -6 | 1 | 1 | 1154 | -1148 | 0 | 2 | 1 | 1946 | -1912 |
| 15 | 1 | 0 | 211 | -219 | 6 | 4 | 0 | 1137 | 1126 | 2 | 8 | 0 | 124 | 129 | -5 | 1 | 1 | 484 | -492 | 1 | 2 | 1 | 210 | -201 |
| 16 | 1 | 0 | 176 | 184 | 7 | 4 | 0 | 342 | -325 | 3 | 8 | 0 | 332 | -328 | -4 | 1 | 1 | 601 | -607 | 2 | 2 | 1 | 649 | -624 |
| 0 | 2 | 0 | 1167 | -1162 | 8 | 4 | 0 | 402 | 403 | 5 | 8 | 0 | 400 | -393 | -3 | 1 | 1 | 410 | 419 | 3 | 2 | 1 | 187 | 185 |
| 1 | 2 | 0 | 1032 | -1013 | 9 | 4 | 0 | 520 | -520 | 6 | 8 | 0 | 161 | -126 | -2 | 1 | 1 | 181 | -185 | 4 | 2 | 1 | 1247 | 1262 |
| 2 | 2 | 0 | 156 | 148 | 10 | 4 | 0 | 254 | -249 | 7 | 8 | 0 | 132 | 148 | -1 | 1 | 1 | 2179 | 2150 | 5 | 2 | 1 | 273 | 282 |
| 3 | 2 | 0 | 595 | -601 | 11 | 4 | 0 | 272 | -295 | 8 | 8 | 0 | 214 | -184 | 0 | 1 | 1 | 950 | 935 | 6 | 2 | 1 | 1047 | 1042 |
| 4 | 2 | 0 | 168 | 179 | 12 | 4 | 0 | 531 | -528 | 9 | 8 | 0 | 391 | 390 | 2 | 1 | 1 | 857 | 880 | 7 | 2 | 1 | 285 | -305 |
| 5 | 2 | 0 | 923 | -889 | 13 | 4 | 0 | 207 | 205 | 1 | 9 | 0 | 176 | 152 | 3 | 1 | 1 | 524 | -535 | 8 | 2 | 1 | 179 | -198 |
| 6 | 2 | 0 | 206 | 209 | 14 | 4 | 0 | 401 | -387 | 4 | 9 | 0 | 191 | 180 | 4 | 1 | 1 | 655 | 640 | 9 | 2 | 1 | 270 | -274 |
| 7 | 2 | 0 | 198 | 200 | 15 | 4 | 0 | 266 | 270 | 6 | 9 | 0 | 142 | 126 | 5 | 1 | 1 | 1514 | -1544 | 10 | 2 | 1 | 440 | -442 |
| 8 | 2 | 0 | 237 | 232 | 4 | 5 | 0 | 527 | -507 | 7 | 9 | 0 | 191 | -185 | 6 | 1 | 1 | 564 | -572 | 12 | 2 | 1 | 404 | -404 |
| 9 | 2 | 0 | 823 | 824 | 6 | 5 | 0 | 528 | -520 | -15 | 0 | 1 | 405 | 397 | 7 | 1 | 1 | 132 | -146 | 13 | 2 | 1 | 148 | 120 |
| 11 | 2 | 0 | 511 | 517 | 8 | 5 | 0 | 335 | 335 | -13 | 0 | 1 | 278 | -289 | 8 | 1 | 1 | 723 | -740 | 14 | 2 | 1 | 213 | -210 |
| 12 | 2 | 0 | 263 | -255 | 10 | 5 | 0 | 599 | 605 | -11 | 0 | 1 | 893 | -878 | 9 | 1 | 1 | 531 | 547 | 15 | 2 | 1 | 213 | 207 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -16 | 3 | 1 | 372 | 375 | -2 | 4 | 1 | 568 | 561 | -6 | 6 | 1 | 304 | -292 | -8 | 8 | 1 | 309 | 310 | -15 | 1 | 2 | 447 | 444 |
| -13 | 3 | 1 | 166 | 124 | -1 | 4 | 1 | 346 | 346 | -5 | 6 | 1 | 724 | 718 | -7 | 8 | 1 | 223 | 218 | -14 | 1 | 2 | 279 | -280 |
| -12 | 3 | 1 | 606 | -613 | 0 | 4 | 1 | 1151 | 1128 | -3 | 6 | 1 | 350 | 342 | -6 | 8 | 1 | 184 | 191 | -12 | 1 | 2 | 202 | -203 |
| -11 | 3 | 1 | 116 | 110 | 1 | 4 | 1 | 1092 | 1064 | -2 | 6 | 1 | 414 | 400 | -5 | 8 | 1 | 181 | 196 | -11 | 1 | 2 | 552 | -542 |
| -10 | 3 | 1 | 998 | -1009 | 3 | 4 | 1 | 910 | 888 | -1 | 6 | 1 | 376 | -369 | -2 | 8 | 1 | 326 | -318 | -10 | 1 | 2 | 603 | -599 |
| -8 | 3 | 1 | 356 | -346 | 4 | 4 | 1 | 977 | -978 | 0 | 6 | 1 | 475 | 464 | 0 | 8 | 1 | 474 | -468 | -9 | 1 | 2 | 807 | -790 |
| -7 | 3 | 1 | 273 | -272 | 5 | 4 | 1 | 130 | 132 | 1 | 6 | 1 | 497 | -476 | 1 | 8 | 1 | 149 | -112 | -7 | 1 | 2 | 121 | 134 |
| -6 | 3 | 1 | 1417 | 1412 | 6 | 4 | 1 | 570 | -572 | 3 | 6 | 1 | 306 | -297 | 4 | 8 | 1 | 406 | 407 | -6 | 1 | 2 | 1280 | 1270 |
| -5 | 3 | 1 | 517 | -500 | 7 | 4 | 1 | 1009 | -998 | 4 | 6 | 1 | 365 | -354 | 6 | 8 | 1 | 379 | 385 | -5 | 1 | 2 | 674 | 663 |
| -4 | 3 | 1 | 1679 | 1636 | 8 | 4 | 1 | 262 | 272 | 5 | 6 | 1 | 196 | -193 | 9 | 8 | 1 | 134 | 156 | -4 | 1 | 2 | 949 | 920 |
| -2 | 3 | 1 | 414 | 390 | 9 | 4 | 1 | 842 | -837 | 6 | 6 | 1 | 313 | -296 | -7 | 9 | 1 | 139 | -157 | -3 | 1 | 2 | 667 | 649 |
| -1 | 3 | 1 | 1046 | 999 | 10 | 4 | 1 | 367 | 375 | 7 | 6 | 1 | 522 | 504 | -6 | 9 | 1 | 211 | 206 | -2 | 1 | 2 | 496 | -491 |
| 0 | 3 | 1 | 2189 | -2131 | 12 | 4 | 1 | 217 | 221 | 9 | 6 | 1 | 487 | 487 | -4 | 9 | 1 | 145 | 143 | -1 | 1 | 2 | 734 | 714 |
| 1 | 3 | 1 | 358 | 348 | 13 | 4 | 1 | 535 | 531 | 10 | 6 | 1 | 201 | 201 | -3 | 9 | 1 | 139 | 146 | 0 | 1 | 2 | 920 | -920 |
| 2 | 3 | 1 | 1244 | -1242 | 15 | 4 | 1 | 371 | 369 | 12 | 6 | 1 | 174 | 165 | -1 | 9 | 1 | 249 | 250 | 1 | 1 | 2 | 532 | -532 |
| 3 | 3 | 1 | 473 | -456 | -13 | 5 | 1 | 502 | -473 | 13 | 6 | 1 | 313 | -303 | 0 | 9 | 1 | 238 | -238 | 2 | 1 | 2 | 899 | -903 |
| 4 | 3 | 1 | 529 | -544 | -11 | 5 | 1 | 246 | 245 | -12 | 7 | 1 | 350 | 334 | 2 | 9 | 1 | 113 | -79 | 3 | 1 | 2 | 884 | -896 |
| 5 | 3 | 1 | 673 | -665 | -9 | 5 | 1 | 577 | 574 | -10 | 7 | 1 | 605 | 596 | 4 | 9 | 1 | 117 | -101 | 4 | 1 | 2 | 502 | -456 |
| 6 | 3 | 1 | 948 | 946 | -8 | 5 | 1 | 157 | 148 | -8 | 7 | 1 | 204 | 193 | 5 | 9 | 1 | 158 | -162 | 5 | 1 | 2 | 752 | -759 |
| 8 | 3 | 1 | 1159 | 1192 | -7 | 5 | 1 | 336 | 329 | -7 | 7 | 1 | 160 | -156 | -16 | 0 | 2 | 646 | -642 | 6 | 1 | 2 | 1198 | 1192 |
| 9 | 3 | 1 | 294 | 298 | -3 | 5 | 1 | 326 | -319 | -6 | 7 | 1 | 689 | -683 | -14 | 0 | 2 | 711 | -692 | 7 | 1 | 2 | 381 | 381 |
| 10 | 3 | 1 | 473 | 471 | -2 | 5 | 1 | 214 | -214 | -5 | 7 | 1 | 165 | -164 | -12 | 0 | 2 | 510 | 516 | 8 | 1 | 2 | 750 | 768 |
| 11 | 3 | 1 | 168 | 193 | -1 | 5 | 1 | 944 | -931 | -4 | 7 | 1 | 899 | -893 | -10 | 0 | 2 | 988 | 984 | 9 | 1 | 2 | 657 | 665 |
| 12 | 3 | 1 | 580 | -585 | 2 | 5 | 1 | 132 | 127 | -3 | 7 | 1 | 134 | 138 | -8 | 0 | 2 | 1056 | 1054 | 11 | 1 | 2 | 325 | 328 |
| 14 | 3 | 1 | 666 | -652 | 3 | 5 | 1 | 273 | 264 | -2 | 7 | 1 | 200 | -179 | -6 | 0 | 2 | 142 | -165 | 12 | 1 | 2 | 568 | -576 |
| 15 | 3 | 1 | 123 | -71 | 5 | 5 | 1 | 711 | 703 | -1 | 7 | 1 | 477 | 468 | -4 | 0 | 2 | 1727 | -1696 | 14 | 1 | 2 | 537 | -534 |
| -15 | 4 | 1 | 214 | -227 | 6 | 5 | 1 | 132 | -109 | 0 | 7 | 1 | 871 | 870 | -2 | 0 | 2 | 1664 | -1580 | 15 | 1 | 2 | 294 | -300 |
| -14 | 4 | 1 | 267 | 268 | 7 | 5 | 1 | 176 | 177 | 2 | 7 | 1 | 698 | 691 | 0 | 0 | 2 | 110 | -122 | -16 | 2 | 2 | 265 | 264 |
| -13 | 4 | 1 | 278 | 268 | 9 | 5 | 1 | 164 | -165 | 3 | 7 | 1 | 156 | -120 | 2 | 0 | 2 | 680 | 734 | -14 | 2 | 2 | 278 | 275 |
| -11 | 4 | 1 | 635 | 640 | 11 | 5 | 1 | 304 | -295 | 4 | 7 | 1 | 235 | 242 | 4 | 0 | 2 | 2935 | 3056 | -13 | 2 | 2 | 565 | 577 |
| -9 | 4 | 1 | 579 | 570 | 13 | 5 | 1 | 292 | -300 | 5 | 7 | 1 | 280 | -269 | 6 | 0 | 2 | 264 | 247 | -12 | 2 | 2 | 322 | -326 |
| -8 | 4 | 1 | 405 | -409 | -13 | 6 | 1 | 227 | -222 | 6 | 7 | 1 | 478 | -456 | 8 | 0 | 2 | 1620 | -1683 | -11 | 2 | 2 | 392 | 391 |
| -7 | 4 | 1 | 476 | -466 | -11 | 6 | 1 | 413 | -392 | 8 | 7 | 1 | 648 | -635 | 10 | 0 | 2 | 1168 | -1189 | -10 | 2 | 2 | 365 | -372 |
| -6 | 4 | 1 | 458 | -466 | -9 | 6 | 1 | 347 | -340 | 9 | 7 | 1 | 169 | 161 | 12 | 0 | 2 | 127 | 84 | -7 | 2 | 2 | 906 | -909 |
| -5 | 4 | 1 | 1208 | -1184 | -8 | 6 | 1 | 186 | -178 | 10 | 7 | 1 | 229 | -241 | 14 | 0 | 2 | 568 | 577 | -6 | 2 | 2 | 319 | 314 |
| -3 | 4 | 1 | 659 | -632 | -7 | 6 | 1 | 290 | 295 | 11 | 7 | 1 | 142 | 157 | 16 | 0 | 2 | 426 | 426 | -5 | 2 | 2 | 886 | -903 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 4 | 3 | 9 | 250 | -264 | 5 | 5 | 9 | 882 | 879 | -10 | 0 | 10 | 568 | 584 | -8 | 2 | 10 | 251 | -264 | -10 | 4 | 10 | 357 | -371 |
| 5 | 3 | 9 | 265 | -281 | 7 | 5 | 9 | 176 | 184 | -8 | 0 | 10 | 283 | 268 | -7 | 2 | 10 | 450 | -410 | -8 | 4 | 10 | 318 | -318 |
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| 7 | 3 | 9 | 262 | -289 | 11 | 5 | 9 | 354 | -341 | -2 | 0 | 10 | 411 | -395 | -4 | 2 | 10 | 219 | 226 | -4 | 4 | 10 | 294 | 293 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| -4 | 6 | 10 | 283 | -297 | -6 | 1 | 11 | 376 | 367 | -12 | 3 | 11 | 210 | 219 | -1 | 5 | 11 | 183 | 181 | -10 | 1 | 12 | 415 | -394 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
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| 9 | 3 | 12 | 383 | 372 | -11 | 0 | 13 | 478 | 466 | 0 | 2 | 13 | 779 | 784 | 3 | 5 | 13 | 172 | -178 | -5 | 2 | 14 | 572 | 582 |
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| 2 | 4 | 12 | 320 | 322 | -10 | 1 | 13 | 266 | -262 | -4 | 3 | 13 | 438 | -444 | -6 | 0 | 14 | 327 | -304 | -9 | 3 | 14 | 257 | -260 |
| 3 | 4 | 12 | 387 | -384 | -9 | 1 | 13 | 210 | 197 | -3 | 3 | 13 | 160 | -187 | -4 | 0 | 14 | 312 | 308 | -8 | 3 | 14 | 134 | -131 |
| 4 | 4 | 12 | 145 | 135 | -8 | 1 | 13 | 258 | -256 | -2 | 3 | 13 | 467 | -458 | -2 | 0 | 14 | 657 | 644 | -7 | 3 | 14 | 343 | -334 |
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| 9 | 4 | 12 | 219 | 193 | -3 | 1 | 13 | 400 | -396 | 8 | 3 | 13 | 283 | -293 | 6 | 0 | 14 | 173 | -168 | -1 | 3 | 14 | 477 | 468 |
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| -4 | 5 | 12 | 392 | -423 | 0 | 1 | 13 | 101 | -82 | -6 | 4 | 13 | 243 | 240 | -8 | 1 | 14 | 130 | -132 | 3 | 3 | 14 | 288 | -282 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 5 | 3 | 14 | 347 | -338 | -7 | 0 | 15 | 511 | 491 | 4 | 1 | 15 | 102 | -104 | 0 | 3 | 15 | 319 | -336 | -2 | 1 | 16 | 323 | -329 |
| 6 | 3 | 14 | 152 | -136 | -5 | 0 | 15 | 355 | 345 | 5 | 1 | 15 | 127 | -127 | 1 | 3 | 15 | 178 | -152 | 0 | 1 | 16 | 159 | -155 |
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| -4 | 4 | 14 | 187 | -203 | 5 | 0 | 15 | 329 | 323 | -5 | 2 | 15 | 140 | -125 | -2 | 4 | 15 | 178 | 166 | -3 | 2 | 16 | 375 | 393 |
| -2 | 4 | 14 | 378 | -379 | 7 | 0 | 15 | 476 | 470 | -4 | 2 | 15 | 519 | -522 | -1 | 4 | 15 | 226 | 235 | -1 | 2 | 16 | 367 | 361 |
| -1 | 4 | 14 | 141 | 157 | -9 | 1 | 15 | 262 | -259 | -2 | 2 | 15 | 448 | -463 | 1 | 4 | 15 | 318 | 319 | 2 | 2 | 16 | 194 | -188 |
| 0 | 4 | 14 | 257 | -250 | -8 | 1 | 15 | 146 | -141 | -1 | 2 | 15 | 178 | 149 | 2 | 4 | 15 | 237 | -256 | 3 | 2 | 16 | 286 | -266 |
| 1 | 4 | 14 | 194 | 197 | -6 | 1 | 15 | 313 | -298 | 1 | 2 | 15 | 128 | 128 | -6 | 0 | 16 | 312 | -296 | -4 | 3 | 16 | 165 | -153 |
| 2 | 4 | 14 | 164 | 184 | -5 | 1 | 15 | 307 | 309 | 2 | 2 | 15 | 607 | 586 | -4 | 0 | 16 | 477 | -447 | -3 | 3 | 16 | 207 | -218 |
| 4 | 4 | 14 | 263 | 267 | -4 | 1 | 15 | 149 | -150 | 4 | 2 | 15 | 412 | 431 | 0 | 0 | 16 | 299 | 315 | 1 | 3 | 16 | 455 | 445 |
| 6 | 4 | 14 | 196 | 208 | -3 | 1 | 15 | 526 | 517 | 5 | 2 | 15 | 124 | -115 | 2 | 0 | 16 | 461 | 458 | -3 | 0 | 17 | 394 | 379 |
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| 0 | 5 | 14 | 359 | -364 | 1 | 1 | 15 | 421 | -436 | -6 | 3 | 15 | 412 | 394 | -5 | 1 | 16 | 234 | 227 | 1 | 0 | 17 | 175 | -220 |
| 2 | 5 | 14 | 508 | -507 | 2 | 1 | 15 | 282 | 270 | -5 | 3 | 15 | 185 | 173 | -4 | 1 | 16 | 274 | -265 | -2 | 1 | 17 | 202 | -212 |
| -9 | 0 | 15 | 125 | 145 | 3 | 1 | 15 | 285 | -299 | -2 | 3 | 15 | 247 | -248 | -3 | 1 | 16 | 300 | 295 | -1 | 1 | 17 | 184 | 211 |

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Dimethyldithiocarbamato-2-(2-pyridyl)phenyltellurium(II) PAGE 1

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 0 | 0 | 759 | 908 | 1 | 2 | 0 | 225 | -195 | 4 | 4 | 0 | 121 | 132 | 10 | 6 | 0 | 102 | -101 | 1 | 10 | 0 | 233 | -258 |
| 2 | 0 | 0 | 777 | 883 | 2 | 2 | 0 | 245 | -242 | 5 | 4 | 0 | 183 | 187 | -4 | 7 | 0 | 121 | -128 | 2 | 10 | 0 | 169 | -185 |
| 3 | 0 | 0 | 646 | 688 | 3 | 2 | 0 | 591 | -649 | 6 | 4 | 0 | 417 | 432 | -3 | 7 | 0 | 68 | -26 | 3 | 10 | 0 | 246 | -261 |
| 4 | 0 | 0 | 200 | -191 | 4 | 2 | 0 | 296 | -320 | 7 | 4 | 0 | 455 | 478 | -2 | 7 | 0 | 67 | -64 | 4 | 10 | 0 | 203 | -213 |
| 5 | 0 | 0 | 44 | 39 | 5 | 2 | 0 | 558 | -598 | 8 | 4 | 0 | 268 | 272 | -1 | 7 | 0 | 406 | 424 | 5 | 10 | 0 | 80 | -83 |
| 6 | 0 | 0 | 367 | -376 | 6 | 2 | 0 | 380 | -406 | 9 | 4 | 0 | 282 | 270 | 0 | 7 | 0 | 459 | 467 | 6 | 10 | 0 | 67 | -73 |
| 7 | 0 | 0 | 220 | -215 | 7 | 2 | 0 | 142 | -151 | 10 | 4 | 0 | 133 | 124 | 1 | 7 | 0 | 397 | 405 | -7 | 10 | 1 | 154 | -163 |
| 8 | 0 | 0 | 225 | -217 | 8 | 2 | 0 | 188 | -182 | -6 | 5 | 0 | 211 | -212 | 2 | 7 | 0 | 592 | 559 | -6 | 10 | 1 | 121 | -125 |
| 9 | 0 | 0 | 191 | -175 | 9 | 2 | 0 | 33 | -28 | -5 | 5 | 0 | 204 | -206 | 3 | 7 | 0 | 264 | 255 | -5 | 10 | 1 | 56 | -60 |
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| 6 | 1 | 0 | 499 | -525 | 6 | 3 | 0 | 26 | -20 | 10 | 5 | 0 | 136 | 122 | 6 | 8 | 0 | 338 | -345 | -6 | -8 | 1 | 199 | 209 |
| 7 | 1 | 0 | 468 | -489 | 8 | 3 | 0 | 80 | 90 | -5 | 6 | 0 | 215 | -210 | 7 | 8 | 0 | 333 | -357 | -5 | -8 | 1 | 330 | 331 |
| 8 | 1 | 0 | 286 | -278 | 9 | 3 | 0 | 71 | 75 | -4 | 6 | 0 | 63 | -59 | 8 | 8 | 0 | 274 | -292 | -4 | -8 | 1 | 293 | 275 |
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| -8 | 2 | 0 | 212 | 198 | -6 | 4 | 0 | 107 | 110 | -1 | 6 | 0 | 188 | 200 | -1 | 9 | 0 | 96 | 103 | -2 | -8 | 1 | 291 | 270 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 5 | -6 | 1 | 57 | 52 | -7 | -3 | 1 | 316 | -329 | -5 | -1 | 1 | 404 | 428 | -1 | 1 | 1 | 1147 | -1183 | -1 | 3 | 1 | 325 | -306 |
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| -1 | -5 | 1 | 630 | -549 | 3 | -3 | 1 | 290 | 323 | 5 | -1 | 1 | 551 | -593 | 8 | 1 | 1 | 103 | 98 | 8 | 3 | 1 | 161 | 154 |
| 0 | -5 | 1 | 498 | -467 | 4 | -3 | 1 | 134 | 137 | 6 | -1 | 1 | 523 | -548 | 9 | 1 | 1 | 199 | 178 | 9 | 3 | 1 | 74 | 71 |
| 1 | -5 | 1 | 85 | -84 | 5 | -3 | 1 | 233 | 244 | 7 | -1 | 1 | 266 | -261 | -8 | 2 | 1 | 81 | 78 | 10 | 3 | 1 | 70 | -68 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 2 | 5 | 1 | 89 | 91 | 7 | 7 | 1 | 168 | -184 | -7 | -9 | 2 | 246 | 259 | -3 | -6 | 2 | 337 | -305 | -9 | -3 | 2 | 77 | 71 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 4 | 8 | 2 | 549 | 501 | -4 | -8 | 3 | 150 | -144 | -4 | -5 | 3 | 322 | 317 | 1 | -3 | 3 | 164 | -180 | 3 | -1 | 3 | 923 | 976 |
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 1 | 7 | 4 | 356 | -347 | -5 | -9 | 5 | 173 | 176 | -6 | -5 | 5 | 217 | -223 | 4 | -3 | 5 | 317 | -328 | -5 | 0 | 5 | 257 | -256 |
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| 0 | 8 | 4 | 187 | -182 | -6 | -7 | 5 | 114 | -112 | -7 | -4 | 5 | 104 | -100 | -1 | -2 | 5 | 388 | -373 | 6 | 0 | 5 | 424 | 436 |
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| 3 | 8 | 4 | 345 | -324 | -3 | -7 | 5 | 329 | -335 | -4 | -4 | 5 | 215 | 210 | 2 | -2 | 5 | 450 | -434 | -8 | 1 | 5 | 122 | -114 |
| 4 | 8 | 4 | 450 | -421 | -2 | -7 | 5 | 320 | -329 | -3 | -4 | 5 | 452 | 451 | 3 | -2 | 5 | 429 | -432 | -7 | 1 | 5 | 335 | -326 |
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| 2 | 9 | 4 | 166 | -164 | -6 | -6 | 5 | 326 | -327 | 5 | -4 | 5 | 144 | -139 | -3 | -1 | 5 | 279 | -278 | 1 | 1 | 5 | 254 | 245 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 7 | 1 | 5 | 267 | 274 | -4 | 4 | 5 | 208 | 207 | -3 | 7 | 5 | 156 | -159 | -2 | -8 | 6 | 57 | 57 | -6 | -4 | 6 | 154 | 156 |
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| 2 | 2 | 5 | 591 | 574 | 9 | 4 | 5 | 151 | -148 | 8 | 7 | 5 | 349 | 367 | -6 | -6 | 6 | 72 | 77 | 5 | -4 | 6 | 37 | -39 |
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| 4 | 2 | 5 | 165 | 157 | -4 | 5 | 5 | 258 | 262 | -2 | 8 | 5 | 88 | -89 | -4 | -6 | 6 | 355 | 360 | -7 | -3 | 6 | 116 | 116 |
| 5 | 2 | 5 | 80 | 85 | -1 | 5 | 5 | 157 | -156 | 0 | 8 | 5 | 35 | -35 | -3 | -6 | 6 | 210 | 218 | -6 | -3 | 6 | 83 | 84 |
| 6 | 2 | 5 | 131 | -130 | 0 | 5 | 5 | 365 | -365 | 1 | 8 | 5 | 114 | 109 | -2 | -6 | 6 | 304 | 300 | -5 | -3 | 6 | 159 | -161 |
| 7 | 2 | 5 | 123 | -126 | 1 | 5 | 5 | 301 | -303 | 2 | 8 | 5 | 54 | 54 | -1 | -6 | 6 | 255 | 246 | -4 | -3 | 6 | 208 | -209 |
| 8 | 2 | 5 | 107 | -109 | 2 | 5 | 5 | 554 | -527 | 3 | 8 | 5 | 159 | 165 | 0 | -6 | 6 | 162 | 155 | -3 | -3 | 6 | 468 | -473 |
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| -7 | 3 | 5 | 58 | -56 | 4 | 5 | 5 | 253 | -251 | 5 | 8 | 5 | 228 | 221 | 2 | -6 | 6 | 93 | -98 | -1 | -3 | 6 | 449 | -457 |
| -5 | 3 | 5 | 45 | -43 | 5 | 5 | 5 | 314 | -319 | 6 | 8 | 5 | 374 | 363 | 3 | -6 | 6 | 111 | -114 | 0 | -3 | 6 | 672 | -638 |
| -4 | 3 | 5 | 168 | 170 | 6 | 5 | 5 | 131 | -136 | 7 | 8 | 5 | 148 | 153 | -8 | -5 | 6 | 175 | 171 | 1 | -3 | 6 | 242 | -240 |
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| 0 | 3 | 5 | 432 | 428 | -2 | 6 | 5 | 206 | -206 | 1 | 9 | 5 | 221 | 237 | -4 | -5 | 6 | 227 | 233 | 6 | -3 | 6 | 220 | 217 |
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| 2 | 3 | 5 | 134 | 153 | 0 | 6 | 5 | 540 | -537 | 3 | 9 | 5 | 220 | 227 | -2 | -5 | 6 | 212 | 206 | -6 | -2 | 6 | 228 | -223 |
| 3 | 3 | 5 | 39 | 42 | 1 | 6 | 5 | 624 | -605 | 4 | 9 | 5 | 235 | 225 | -1 | -5 | 6 | 196 | 193 | -5 | -2 | 6 | 118 | -118 |
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| 6 | 3 | 5 | 301 | -318 | 3 | 6 | 5 | 488 | -457 | 8 | 9 | 5 | 81 | -96 | 1 | -5 | 6 | 173 | -165 | -3 | -2 | 6 | 315 | -318 |
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| 8 | 3 | 5 | 322 | -325 | 6 | 6 | 5 | 122 | 127 | 5 | 10 | 5 | 66 | -48 | 3 | -5 | 6 | 271 | -268 | -1 | -2 | 6 | 279 | -292 |
| 9 | 3 | 5 | 158 | -153 | 7 | 6 | 5 | 285 | 290 | 6 | 10 | 5 | 127 | -123 | 4 | -5 | 6 | 248 | -250 | 0 | -2 | 6 | 194 | -178 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 6 | -2 | 6 | 221 | 220 | -3 | 1 | 6 | 422 | 427 | 3 | 3 | 6 | 408 | -407 | 2 | 6 | 6 | 411 | 414 | 4 | 10 | 6 | 112 | -115 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 0 | -2 | 7 | 432 | 425 | -7 | 1 | 7 | 280 | 275 | 3 | 3 | 7 | 565 | 554 | 1 | 6 | 7 | 150 | 147 | -6 | -6 | 8 | 249 | -251 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -5 | -6 | 8 | 134 | -149 | 3 | -3 | 8 | 279 | -268 | 2 | 0 | 8 | 39 | 41 | 1 | 3 | 8 | 358 | 359 | 3 | 6 | 8 | 270 | -274 |
| -4 | -6 | 8 | 172 | -172 | 4 | -3 | 8 | 218 | -214 | 3 | 0 | 8 | 57 | 42 | 2 | 3 | 8 | 34 | -40 | 4 | 6 | 8 | 141 | -133 |
| -3 | -6 | 8 | 129 | -138 | 5 | -3 | 8 | 135 | -132 | 4 | 0 | 8 | 151 | 149 | 3 | 3 | 8 | 194 | 198 | 5 | 6 | 8 | 115 | -114 |
| -2 | -6 | 8 | 72 | -78 | -7 | -2 | 8 | 152 | 148 | 5 | 0 | 8 | 192 | 181 | 4 | 3 | 8 | 55 | 46 | 6 | 6 | 8 | 81 | 81 |
| -1 | -6 | 8 | 48 | 47 | -6 | -2 | 8 | 128 | 131 | 6 | 0 | 8 | 163 | 163 | 5 | 3 | 8 | 106 | -109 | 7 | 6 | 8 | 65 | 62 |
| 0 | -6 | 8 | 159 | 147 | -5 | -2 | 8 | 145 | 146 | 7 | 0 | 8 | 121 | 124 | 7 | 3 | 8 | 215 | -218 | 8 | 6 | 8 | 142 | 144 |
| 1 | -6 | 8 | 130 | 125 | -4 | -2 | 8 | 176 | 183 | -6 | 1 | 8 | 120 | -123 | 8 | 3 | 8 | 154 | -150 | -1 | 7 | 8 | 208 | -215 |
| -7 | -5 | 8 | 60 | -68 | -2 | -2 | 8 | 82 | 77 | -5 | 1 | 8 | 219 | -216 | -4 | 4 | 8 | 150 | 141 | 1 | 7 | 8 | 43 | -40 |
| -6 | -5 | 8 | 59 | -68 | -1 | -2 | 8 | 154 | -149 | -4 | 1 | 8 | 260 | -267 | -3 | 4 | 8 | 317 | 315 | 3 | 7 | 8 | 71 | 51 |
| -3 | -5 | 8 | 51 | -46 | 0 | -2 | 8 | 334 | -336 | -3 | 1 | 8 | 243 | -245 | -2 | 4 | 8 | 206 | 206 | 4 | 7 | 8 | 84 | 69 |
| -2 | -5 | 8 | 146 | 143 | 1 | -2 | 8 | 344 | -337 | -2 | 1 | 8 | 290 | -289 | -1 | 4 | 8 | 190 | 190 | 5 | 7 | 8 | 149 | 135 |
| -1 | -5 | 8 | 165 | 163 | 2 | -2 | 8 | 430 | -425 | -1 | 1 | 8 | 46 | 45 | 0 | 4 | 8 | 42 | 44 | 6 | 7 | 8 | 93 | 78 |
| 0 | -5 | 8 | 302 | 299 | 3 | -2 | 8 | 339 | -337 | 1 | 1 | 8 | 282 | 278 | 2 | 4 | 8 | 129 | -126 | 7 | 7 | 8 | 138 | 139 |
| 1 | -5 | 8 | 270 | 257 | 4 | -2 | 8 | 298 | -289 | 2 | 1 | 8 | 389 | 389 | 3 | 4 | 8 | 364 | -366 | 0 | 8 | 8 | 59 | -55 |
| 2 | -5 | 8 | 203 | 202 | 5 | -2 | 8 | 136 | -136 | 3 | 1 | 8 | 319 | 316 | 4 | 4 | 8 | 135 | -131 | 3 | 8 | 8 | 263 | 277 |
| 3 | -5 | 8 | 246 | 247 | -7 | -1 | 8 | 38 | 36 | 4 | 1 | 8 | 574 | 565 | 5 | 4 | 8 | 286 | -287 | 4 | 8 | 8 | 251 | 248 |
| -7 | -4 | 8 | 54 | -52 | -6 | -1 | 8 | 48 | 48 | 5 | 1 | 8 | 276 | 275 | 6 | 4 | 8 | 248 | -246 | 5 | 8 | 8 | 201 | 211 |
| -6 | -4 | 8 | 146 | 145 | -5 | -1 | 8 | 59 | -55 | 6 | 1 | 8 | 161 | 156 | 7 | 4 | 8 | 37 | -46 | 6 | 8 | 8 | 197 | 206 |
| -5 | -4 | 8 | 146 | 144 | -4 | -1 | 8 | 137 | -121 | 7 | 1 | 8 | 68 | 67 | 8 | 4 | 8 | 135 | -140 | 7 | 8 | 8 | 46 | 51 |
| -4 | -4 | 8 | 200 | 201 | -3 | -1 | 8 | 30 | 24 | -6 | 2 | 8 | 48 | -35 | -4 | 5 | 8 | 119 | 121 | 3 | 9 | 8 | 175 | 187 |
| -3 | -4 | 8 | 360 | 365 | -2 | -1 | 8 | 237 | -241 | -4 | 2 | 8 | 150 | -147 | -3 | 5 | 8 | 125 | 130 | 4 | 9 | 8 | 209 | 216 |
| -2 | -4 | 8 | 248 | 252 | -1 | -1 | 8 | 219 | -219 | -1 | 2 | 8 | 98 | 103 | -2 | 5 | 8 | 167 | 167 | 5 | 9 | 8 | 128 | 137 |
| -1 | -4 | 8 | 302 | 298 | 0 | -1 | 8 | 253 | -253 | 0 | 2 | 8 | 353 | 356 | 0 | 5 | 8 | 42 | -47 | -5 | -6 | 9 | 152 | 161 |
| 0 | -4 | 8 | 110 | 109 | 1 | -1 | 8 | 370 | -366 | 1 | 2 | 8 | 199 | 208 | 1 | 5 | 8 | 239 | -245 | -4 | -6 | 9 | 201 | 208 |
| 1 | -4 | 8 | 75 | 70 | 2 | -1 | 8 | 121 | -117 | 2 | 2 | 8 | 460 | 463 | 2 | 5 | 8 | 303 | -308 | -3 | -6 | 9 | 169 | 177 |
| 2 | -4 | 8 | 76 | 69 | 3 | -1 | 8 | 275 | -264 | 3 | 2 | 8 | 440 | 432 | 3 | 5 | 8 | 393 | -400 | -2 | -6 | 9 | 180 | 179 |
| 3 | -4 | 8 | 53 | -48 | 4 | -1 | 8 | 129 | -126 | 4 | 2 | 8 | 207 | 199 | 4 | 5 | 8 | 441 | -433 | -1 | -6 | 9 | 94 | 100 |
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| -6 | -3 | 8 | 139 | 133 | -6 | 0 | 8 | 129 | -130 | 6 | 2 | 8 | 44 | -41 | 6 | 5 | 8 | 196 | -198 | -6 | -5 | 9 | 145 | 139 |
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| -4 | -3 | 8 | 261 | 269 | -4 | 0 | 8 | 301 | -308 | 8 | 2 | 8 | 117 | -118 | -3 | 6 | 8 | 44 | -55 | -4 | -5 | 9 | 94 | 179 |
| -3 | -3 | 8 | 307 | 310 | -3 | 0 | 8 | 420 | -431 | -4 | 3 | 8 | 240 | 236 | -2 | 6 | 8 | 106 | -110 | -3 | -5 | 9 | 118 | 119 |
| -2 | -3 | 8 | 305 | 310 | -2 | 0 | 8 | 369 | -368 | -3 | 3 | 8 | 181 | 180 | -1 | 6 | 8 | 76 | -77 | -1 | -5 | 9 | 55 | -56 |
| -1 | -3 | 8 | 98 | 99 | -1 | 0 | 8 | 331 | -349 | -2 | 3 | 8 | 396 | 405 | 0 | 6 | 8 | 261 | -270 | 0 | -5 | 9 | 134 | -130 |
| 1 | -3 | 8 | 150 | -138 | 0 | 0 | 8 | 155 | -156 | -1 | 3 | 8 | 241 | 239 | 1 | 6 | 8 | 135 | -143 | 1 | -5 | 9 | 113 | -103 |
| 2 | -3 | 8 | 158 | -140 | 1 | 0 | 8 | 110 | 104 | 0 | 3 | 8 | 117 | 113 | 2 | 6 | 8 | 236 | -246 | 2 | -5 | 9 | 201 | -191 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -6 | -4 | 9 | 146 | 150 | 1 | -1 | 9 | 321 | 311 | 3 | 2 | 9 | 245 | -239 | 6 | 5 | 9 | 181 | 174 | 2 | -3 | 10 | 196 | 189 |
| -5 | -4 | 9 | 211 | 218 | 2 | -1 | 9 | 252 | 248 | 4 | 2 | 9 | 336 | -331 | 7 | 5 | 9 | 159 | 161 | 3 | -3 | 10 | 120 | 114 |
| -4 | -4 | 9 | 76 | 84 | 3 | -1 | 9 | 350 | 341 | 5 | 2 | 9 | 178 | -174 | -2 | 6 | 9 | 161 | -151 | -3 | -2 | 10 | 179 | 183 |
| -3 | -4 | 9 | 65 | 70 | 4 | -1 | 9 | 171 | 167 | 6 | 2 | 9 | 104 | -108 | 0 | 6 | 9 | 125 | 131 | -2 | -2 | 10 | 255 | 255 |
| -2 | -4 | 9 | 117 | -111 | 5 | -1 | 9 | 122 | 119 | 7 | 2 | 9 | 139 | -139 | 1 | 6 | 9 | 95 | 102 | -1 | -2 | 10 | 223 | 227 |
| -1 | -4 | 9 | 235 | -238 | -6 | 0 | 9 | 41 | -37 | -5 | 3 | 9 | 106 | 105 | 2 | 6 | 9 | 303 | 320 | 0 | -2 | 10 | 374 | 369 |
| 0 | -4 | 9 | 213 | -207 | -5 | 0 | 9 | 118 | -118 | -4 | 3 | 9 | 140 | 141 | 3 | 6 | 9 | 240 | 247 | 1 | -2 | 10 | 234 | 229 |
| 1 | -4 | 9 | 287 | -265 | -2 | 0 | 9 | 152 | 153 | -3 | 3 | 9 | 103 | 96 | 4 | 6 | 9 | 235 | 240 | 2 | -2 | 10 | 175 | 170 |
| 2 | -4 | 9 | 238 | -231 | -1 | 0 | 9 | 311 | 317 | -2 | 3 | 9 | 102 | -105 | 5 | 6 | 9 | 174 | 179 | 3 | -2 | 10 | 88 | 87 |
| 3 | -4 | 9 | 208 | -195 | 0 | 0 | 9 | 213 | 213 | -1 | 3 | 9 | 69 | -80 | 7 | 6 | 9 | 97 | 97 | 4 | -2 | 10 | 47 | -50 |
| -6 | -3 | 9 | 57 | -61 | 1 | 0 | 9 | 329 | 322 | 0 | 3 | 9 | 287 | -290 | 0 | 7 | 9 | 155 | 156 | -5 | -1 | 10 | 45 | 36 |
| -5 | -3 | 9 | 72 | -74 | 2 | 0 | 9 | 252 | 248 | 1 | 3 | 9 | 282 | -277 | 1 | 7 | 9 | 207 | 206 | -4 | -1 | 10 | 145 | 139 |
| -4 | -3 | 9 | 80 | -82 | 3 | 0 | 9 | 280 | 263 | 2 | 3 | 9 | 389 | -387 | 2 | 7 | 9 | 241 | 245 | -3 | -1 | 10 | 97 | 98 |
| -3 | -3 | 9 | 215 | -227 | 4 | 0 | 9 | 116 | 115 | 3 | 3 | 9 | 323 | -321 | 3 | 7 | 9 | 262 | 262 | -2 | -1 | 10 | 163 | 187 |
| -2 | -3 | 9 | 182 | -179 | 5 | 0 | 9 | 61 | -61 | 4 | 3 | 9 | 127 | -124 | 4 | 7 | 9 | 121 | 121 | -1 | -1 | 10 | 160 | 158 |
| -1 | -3 | 9 | 241 | -251 | 6 | 0 | 9 | 52 | -54 | 5 | 3 | 9 | 189 | -189 | 5 | 7 | 9 | 96 | 103 | 1 | -1 | 10 | 194 | 182 |
| 0 | -3 | 9 | 251 | -251 | -6 | 1 | 9 | 46 | 43 | 6 | 3 | 9 | 52 | 54 | 7 | 7 | 9 | 111 | -100 | 3 | -1 | 10 | 85 | -86 |
| 1 | -3 | 9 | 236 | -227 | -5 | 1 | 9 | 152 | 156 | 7 | 3 | 9 | 45 | 51 | 2 | 8 | 9 | 146 | 143 | 4 | -1 | 10 | 104 | -98 |
| 2 | -3 | 9 | 230 | -224 | -4 | 1 | 9 | 95 | 108 | -4 | 4 | 9 | 73 | -74 | 4 | 8 | 9 | 61 | 69 | 5 | -1 | 10 | 214 | -202 |
| 3 | -3 | 9 | 55 | -55 | -3 | 1 | 9 | 276 | 281 | -3 | 4 | 9 | 59 | -64 | 5 | 8 | 9 | 87 | -95 | -5 | 0 | 10 | 167 | 170 |
| -6 | -2 | 9 | 190 | -184 | -2 | 1 | 9 | 147 | 147 | -2 | 4 | 9 | 56 | -61 | -4 | -5 | 10 | 141 | -144 | -4 | 0 | 10 | 181 | 180 |
| -5 | -2 | 9 | 259 | -267 | -1 | 1 | 9 | 197 | 202 | -1 | 4 | 9 | 146 | -151 | -3 | -5 | 10 | 131 | -129 | -3 | 0 | 10 | 137 | 146 |
| -4 | -2 | 9 | 215 | -218 | 0 | 1 | 9 | 284 | 291 | 0 | 4 | 9 | 150 | -161 | -2 | -5 | 10 | 205 | -208 | -2 | 0 | 10 | 63 | -60 |
| -3 | -2 | 9 | 214 | -218 | 2 | 1 | 9 | 415 | 407 | 1 | 4 | 9 | 300 | -304 | -1 | -5 | 10 | 171 | -176 | -1 | 0 | 10 | 31 | -38 |
| -2 | -2 | 9 | 238 | -244 | 3 | 1 | 9 | 65 | 67 | 2 | 4 | 9 | 192 | -195 | 0 | -5 | 10 | 163 | -161 | 0 | 0 | 10 | 210 | -202 |
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| 2 | -2 | 9 | 136 | 135 | 6 | 1 | 9 | 234 | -234 | 4 | 4 | 9 | 151 | -145 | -4 | -4 | 10 | 163 | -169 | 2 | 0 | 10 | 123 | -118 |
| 3 | -2 | 9 | 146 | 140 | 7 | 1 | 9 | 141 | -140 | 5 | 4 | 9 | 93 | 95 | -3 | -4 | 10 | 133 | -140 | 3 | 0 | 10 | 290 | -274 |
| 4 | -2 | 9 | 155 | 151 | -5 | 2 | 9 | 193 | 187 | 6 | 4 | 9 | 68 | 65 | 1 | -4 | 10 | 87 | 82 | 4 | 0 | 10 | 98 | -100 |
| 5 | -2 | 9 | 180 | 175 | -4 | 2 | 9 | 262 | 265 | 7 | 4 | 9 | 182 | 182 | 2 | -4 | 10 | 103 | 103 | 5 | 0 | 10 | 152 | -144 |
| -6 | -1 | 9 | 186 | -179 | -3 | 2 | 9 | 202 | 207 | -3 | 5 | 9 | 211 | -215 | -5 | -3 | 10 | 153 | -156 | -5 | 1 | 10 | 124 | 120 |
| -5 | -1 | 9 | 140 | -140 | -2 | 2 | 9 | 188 | 192 | -2 | 5 | 9 | 135 | -133 | -4 | -3 | 10 | 62 | -58 | -4 | 1 | 10 | 44 | 37 |
| -4 | -1 | 9 | 191 | -184 | -1 | 2 | 9 | 59 | -64 | -1 | 5 | 9 | 80 | -81 | -2 | -3 | 10 | 79 | 77 | -3 | 1 | 10 | 61 | -57 |
| -3 | -1 | 9 | 180 | -174 | 0 | 2 | 9 | 39 | -38 | 0 | 5 | 9 | 138 | -140 | -1 | -3 | 10 | 175 | 176 | -2 | 1 | 10 | 102 | -104 |
| -1 | -1 | 9 | 68 | 67 | 1 | 2 | 9 | 211 | -214 | 4 | 5 | 9 | 130 | 134 | 0 | -3 | 10 | 141 | 129 | -1 | 1 | 10 | 288 | -290 |
| 0 | -1 | 9 | 325 | 326 | 2 | 2 | 9 | 356 | -355 | 5 | 5 | 9 | 139 | 140 | 1 | -3 | 10 | 234 | 226 | 0 | 1 | 10 | 136 | -141 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 1 | 10 | 349 | -343 | 5 | 4 | 10 | 43 | 49 | -1 | -2 | 11 | 135 | -126 | -1 | 2 | 11 | 178 | 186 | -2 | -2 | 12 | 153 | -155 |
| 2 | 1 | 10 | 363 | -360 | 6 | 4 | 10 | 71 | 72 | 0 | -2 | 11 | 109 | -112 | 0 | 2 | 11 | 209 | 214 | -1 | -2 | 12 | 131 | -135 |
| 3 | 1 | 10 | 122 | -116 | -1 | 5 | 10 | 209 | 214 | 1 | -2 | 11 | 133 | -126 | 1 | 2 | 11 | 270 | 266 | 0 | -2 | 12 | 96 | -98 |
| 4 | 1 | 10 | 147 | -150 | 0 | 5 | 10 | 211 | 215 | 2 | -2 | 11 | 194 | -187 | 2 | 2 | 11 | 220 | 216 | 0 | -1 | 12 | 33 | 37 |
| 5 | 1 | 10 | 66 | 59 | 1 | 5 | 10 | 239 | 242 | 3 | -2 | 11 | 126 | -123 | 3 | 2 | 11 | 90 | 91 | 1 | -1 | 12 | 92 | 90 |
| 6 | 1 | 10 | 44 | 43 | 2 | 5 | 10 | 289 | 290 | -3 | -1 | 11 | 139 | -138 | 4 | 2 | 11 | 122 | 120 | 2 | -1 | 12 | 179 | 173 |
| -3 | 2 | 10 | 121 | -123 | 3 | 5 | 10 | 140 | 139 | -2 | -1 | 11 | 238 | -238 | 5 | 2 | 11 | 52 | 45 | -2 | 0 | 12 | 144 | 137 |
| -2 | 2 | 10 | 166 | -172 | 4 | 5 | 10 | 108 | 104 | -1 | -1 | 11 | 154 | -156 | -3 | 3 | 11 | 69 | 68 | -1 | 0 | 12 | 64 | 65 |
| -1 | 2 | 10 | 201 | -214 | 5 | 5 | 10 | 65 | -71 | 0 | -1 | 11 | 272 | -271 | -2 | 3 | 11 | 204 | 207 | 0 | 0 | 12 | 154 | 151 |
| 0 | 2 | 10 | 297 | -302 | 6 | 5 | 10 | 83 | -87 | 1 | -1 | 11 | 163 | -157 | -1 | 3 | 11 | 167 | 174 | 1 | 0 | 12 | 143 | 133 |
| 1 | 2 | 10 | 219 | -211 | 0 | 6 | 10 | 166 | 170 | 2 | -1 | 11 | 87 | -83 | 0 | 3 | 11 | 213 | 207 | 2 | 0 | 12 | 67 | 67 |
| 2 | 2 | 10 | 230 | -227 | 1 | 6 | 10 | 122 | 118 | 3 | -1 | 11 | 91 | -93 | 1 | 3 | 11 | 178 | 173 | 3 | 0 | 12 | 127 | 121 |
| 3 | 2 | 10 | 39 | 42 | 2 | 6 | 10 | 37 | 40 | -4 | 0 | 11 | 132 | -126 | 3 | 3 | 11 | 50 | 52 | -2 | 1 | 12 | 88 | 83 |
| 4 | 2 | 10 | 108 | 106 | 3 | 6 | 10 | 44 | 38 | -3 | 0 | 11 | 139 | -145 | 4 | 3 | 11 | 115 | -109 | -1 | 1 | 12 | 144 | 144 |
| 5 | 2 | 10 | 96 | 91 | 4 | 6 | 10 | 151 | -158 | -2 | 0 | 11 | 193 | -193 | 5 | 3 | 11 | 74 | -83 | 0 | 1 | 12 | 196 | 192 |
| 6 | 2 | 10 | 213 | 206 | 5 | 6 | 10 | 81 | -83 | -1 | 0 | 11 | 146 | -143 | -2 | 4 | 11 | 67 | 65 | 1 | 1 | 12 | 40 | 42 |
| -4 | 3 | 10 | 204 | -211 | 6 | 6 | 10 | 212 | -207 | 0 | 0 | 11 | 107 | -103 | -1 | 4 | 11 | 182 | 184 | 3 | 1 | 12 | 72 | -68 |
| -3 | 3 | 10 | 88 | -88 | 1 | 7 | 10 | 44 | -27 | 1 | 0 | 11 | 222 | -207 | 0 | 4 | 11 | 54 | 58 | -2 | 2 | 12 | 156 | 165 |
| -2 | 3 | 10 | 62 | -49 | 2 | 7 | 10 | 131 | -130 | 2 | 0 | 11 | 79 | 75 | 1 | 4 | 11 | 94 | 90 | -1 | 2 | 12 | 106 | 103 |
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| 2 | 3 | 10 | 63 | 63 | 4 | 7 | 10 | 70 | -74 | 4 | 0 | 11 | 104 | 91 | 3 | 4 | 11 | 139 | -136 | 1 | 2 | 12 | 82 | -82 |
| 3 | 3 | 10 | 72 | 75 | 5 | 7 | 10 | 119 | -122 | -4 | 1 | 11 | 61 | -62 | 4 | 4 | 11 | 107 | -107 | 2 | 2 | 12 | 113 | -118 |
| 4 | 3 | 10 | 140 | 142 | -3 | -4 | 11 | 162 | 156 | -3 | 1 | 11 | 81 | -83 | 5 | 4 | 11 | 225 | -213 | 3 | 2 | 12 | 129 | -127 |
| 5 | 3 | 10 | 196 | 192 | -2 | -4 | 11 | 149 | 152 | -2 | 1 | 11 | 57 | -65 | -1 | 5 | 11 | 58 | -64 | 4 | 2 | 12 | 199 | -189 |
| 6 | 3 | 10 | 167 | 160 | -1 | -4 | 11 | 210 | 206 | -1 | 1 | 11 | 44 | -54 | 1 | 5 | 11 | 83 | -89 | 0 | 3 | 12 | 119 | -118 |
| -3 | 4 | 10 | 64 | -54 | -4 | -3 | 11 | 100 | 103 | 0 | 1 | 11 | 128 | -125 | 2 | 5 | 11 | 80 | -81 | 1 | 3 | 12 | 57 | -60 |
| -2 | 4 | 10 | 73 | 74 | -3 | -3 | 11 | 146 | 153 | 2 | 1 | 11 | 101 | 101 | 3 | 5 | 11 | 136 | -139 | 2 | 3 | 12 | 134 | -125 |
| -1 | 4 | 10 | 68 | 70 | -2 | -3 | 11 | 81 | 81 | 3 | 1 | 11 | 145 | 145 | 4 | 5 | 11 | 124 | -126 | 3 | 3 | 12 | 131 | -132 |
| 0 | 4 | 10 | 170 | 169 | -1 | -3 | 11 | 110 | 116 | 4 | 1 | 11 | 171 | 167 | 5 | 5 | 11 | 112 | -113 | 4 | 3 | 12 | 135 | -125 |
| 1 | 4 | 10 | 255 | 257 | 0 | -3 | 11 | 121 | 120 | 5 | 1 | 11 | 175 | 163 | 1 | 6 | 11 | 115 | -118 | 0 | 4 | 12 | 62 | -64 |
| 2 | 4 | 10 | 152 | 154 | 1 | -3 | 11 | 63 | -57 | -3 | 2 | 11 | 90 | 90 | 2 | 6 | 11 | 203 | -202 | 1 | 4 | 12 | 142 | -148 |
| 3 | 4 | 10 | 212 | 216 | -4 | -2 | 11 | 70 | 71 | -2 | 2 | 11 | 78 | 78 | 3 | 6 | 11 | 49 | -57 | 3 | 4 | 12 | 40 | -31 |
| 4 | 4 | 10 | 147 | 146 | -3 | -2 | 11 | 35 | -32 | | | | | | | | | | | | | | | |

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Dimethydithiocarbamato-2-(2-quinolinyl)phenyltellurium(II) PAGE 1

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 0 | 0 | 777 | -751 | 4 | 2 | 0 | 659 | -685 | 7 | 4 | 0 | 264 | 271 | -6 | 7 | 0 | 377 | -377 | 1 | 9 | 0 | 450 | 447 |
| 2 | 0 | 0 | 2593 | -2552 | 5 | 2 | 0 | 576 | 594 | 8 | 4 | 0 | 63 | 68 | -5 | 7 | 0 | 374 | -362 | 2 | 9 | 0 | 674 | 670 |
| 3 | 0 | 0 | 424 | 417 | 6 | 2 | 0 | 214 | 231 | -8 | 5 | 0 | 199 | -212 | -4 | 7 | 0 | 900 | 887 | 3 | 9 | 0 | 310 | -316 |
| 4 | 0 | 0 | 829 | 844 | 7 | 2 | 0 | 252 | -270 | -7 | 5 | 0 | 469 | -467 | -3 | 7 | 0 | 70 | -80 | 4 | 9 | 0 | 167 | -166 |
| 5 | 0 | 0 | 794 | -810 | -8 | 3 | 0 | 231 | 229 | -6 | 5 | 0 | 570 | 577 | -2 | 7 | 0 | 910 | -916 | 5 | 9 | 0 | 264 | 258 |
| 6 | 0 | 0 | 64 | -40 | -7 | 3 | 0 | 159 | 173 | -5 | 5 | 0 | 218 | 210 | -1 | 7 | 0 | 563 | 569 | 6 | 9 | 0 | 51 | -27 |
| 7 | 0 | 0 | 359 | 368 | -6 | 3 | 0 | 332 | -321 | -4 | 5 | 0 | 1364 | -1326 | 0 | 7 | 0 | 781 | 764 | -7 | 10 | 0 | 137 | -141 |
| 8 | 0 | 0 | 179 | -183 | -5 | 3 | 0 | 174 | -155 | -3 | 5 | 0 | 99 | -92 | 1 | 7 | 0 | 1040 | -1068 | -6 | 10 | 0 | 462 | -462 |
| -8 | 1 | 0 | 318 | -328 | -4 | 3 | 0 | 504 | 478 | -2 | 5 | 0 | 2082 | 1999 | 2 | 7 | 0 | 388 | -390 | -5 | 10 | 0 | 491 | 495 |
| -7 | 1 | 0 | 204 | -194 | -3 | 3 | 0 | 423 | -421 | -1 | 5 | 0 | 793 | -780 | 3 | 7 | 0 | 680 | 691 | -4 | 10 | 0 | 521 | 515 |
| -6 | 1 | 0 | 608 | 625 | -2 | 3 | 0 | 1044 | -957 | 0 | 5 | 0 | 901 | -855 | 4 | 7 | 0 | 121 | -120 | -3 | 10 | 0 | 876 | -871 |
| -5 | 1 | 0 | 250 | -247 | -1 | 3 | 0 | 1400 | 1343 | 1 | 5 | 0 | 1344 | 1329 | 5 | 7 | 0 | 302 | -297 | -2 | 10 | 0 | 307 | -307 |
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| -2 | 1 | 0 | 1977 | 1832 | 2 | 3 | 0 | 313 | 300 | 4 | 5 | 0 | 498 | 505 | -8 | 8 | 0 | 228 | -227 | 1 | 10 | 0 | 531 | -531 |
| -1 | 1 | 0 | 1085 | -995 | 3 | 3 | 0 | 1338 | 1367 | 5 | 5 | 0 | 437 | 434 | -7 | 8 | 0 | 167 | 159 | 2 | 10 | 0 | 158 | 162 |
| 1 | 1 | 0 | 2093 | 2144 | 4 | 3 | 0 | 674 | -684 | 6 | 5 | 0 | 448 | -465 | -6 | 8 | 0 | 515 | 506 | 3 | 10 | 0 | 163 | 150 |
| 2 | 1 | 0 | 1205 | -1210 | 5 | 3 | 0 | 439 | -464 | 7 | 5 | 0 | 141 | -142 | -5 | 8 | 0 | 480 | -472 | 4 | 10 | 0 | 473 | -480 |
| 3 | 1 | 0 | 1272 | -1338 | 6 | 3 | 0 | 364 | 387 | -8 | 6 | 0 | 354 | 361 | -4 | 8 | 0 | 396 | -394 | 5 | 10 | 0 | 82 | 80 |
| 4 | 1 | 0 | 684 | 703 | 7 | 3 | 0 | 155 | 165 | -7 | 6 | 0 | 297 | -296 | -3 | 8 | 0 | 677 | 646 | 6 | 10 | 0 | 225 | 232 |
| 5 | 1 | 0 | 336 | 345 | 8 | 3 | 0 | 218 | -227 | -6 | 6 | 0 | 586 | -583 | -1 | 8 | 0 | 881 | -882 | -7 | 11 | 0 | 250 | 242 |
| 6 | 1 | 0 | 478 | -501 | -8 | 4 | 0 | 238 | -234 | -5 | 6 | 0 | 790 | 769 | 0 | 8 | 0 | 432 | 438 | -6 | 11 | 0 | 109 | -113 |
| 7 | 1 | 0 | 83 | 77 | -7 | 4 | 0 | 305 | 303 | -4 | 6 | 0 | 116 | 114 | 1 | 8 | 0 | 420 | 421 | -5 | 11 | 0 | 359 | -357 |
| 8 | 1 | 0 | 241 | 260 | -6 | 4 | 0 | 107 | 87 | -3 | 6 | 0 | 984 | -958 | 2 | 8 | 0 | 619 | -632 | -4 | 11 | 0 | 683 | 690 |
| -8 | 2 | 0 | 199 | 203 | -5 | 4 | 0 | 892 | -877 | -2 | 6 | 0 | 489 | 494 | 3 | 8 | 0 | 203 | -194 | -3 | 11 | 0 | 347 | 351 |
| -7 | 2 | 0 | 377 | -382 | -4 | 4 | 0 | 90 | -87 | -1 | 6 | 0 | 1407 | 1389 | 4 | 8 | 0 | 713 | 718 | -2 | 11 | 0 | 845 | -835 |
| -6 | 2 | 0 | 286 | -294 | -3 | 4 | 0 | 1213 | 1173 | 0 | 6 | 0 | 998 | -1006 | 5 | 8 | 0 | 155 | 158 | -1 | 11 | 0 | 115 | 111 |
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| -1 | 2 | 0 | 286 | 258 | 2 | 4 | 0 | 1241 | -1234 | 5 | 6 | 0 | 420 | 425 | -4 | 9 | 0 | 657 | -637 | 4 | 11 | 0 | 123 | 118 |
| 0 | 2 | 0 | 1252 | -1226 | 3 | 4 | 0 | 355 | 374 | 6 | 6 | 0 | 203 | 202 | -3 | 9 | 0 | 618 | -622 | 5 | 11 | 0 | 270 | -274 |
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| 2 | 2 | 0 | 1019 | 1015 | 5 | 4 | 0 | 538 | -548 | -8 | 7 | 0 | 69 | 40 | -1 | 9 | 0 | 167 | 180 | -5 | 12 | 0 | 78 | -70 |
| 3 | 2 | 0 | 772 | -782 | 6 | 4 | 0 | 309 | -317 | -7 | 7 | 0 | 440 | 435 | 0 | 9 | 0 | 790 | -785 | -4 | 12 | 0 | 238 | -247 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -3 | 12 | 0 | 542 | 535 | -1 | -15 | 1 | 143 | -139 | -5 | -10 | 1 | 227 | 215 | -2 | -7 | 1 | 568 | 565 | -7 | -4 | 1 | 106 | 102 |
| -2 | 12 | 0 | 87 | -97 | 1 | -15 | 1 | 114 | 122 | -4 | -10 | 1 | 138 | 132 | 0 | -7 | 1 | 220 | -223 | -6 | -4 | 1 | 246 | 257 |
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| 0 | 12 | 0 | 141 | 142 | -3 | -14 | 1 | 118 | -115 | -2 | -10 | 1 | 62 | -69 | 2 | -7 | 1 | 189 | -177 | -4 | -4 | 1 | 189 | -192 |
| 1 | 12 | 0 | 465 | 466 | -2 | -14 | 1 | 137 | 144 | -1 | -10 | 1 | 95 | -98 | 4 | -7 | 1 | 157 | 148 | -3 | -4 | 1 | 224 | 217 |
| 2 | 12 | 0 | 255 | -256 | -1 | -14 | 1 | 66 | 61 | 0 | -10 | 1 | 329 | -316 | 5 | -7 | 1 | 187 | -172 | -1 | -4 | 1 | 356 | -334 |
| 3 | 12 | 0 | 258 | -258 | 0 | -14 | 1 | 111 | -115 | 1 | -10 | 1 | 290 | 293 | 6 | -7 | 1 | 110 | -110 | 0 | -4 | 1 | 463 | -444 |
| 4 | 12 | 0 | 299 | 301 | 1 | -14 | 1 | 202 | -194 | 2 | -10 | 1 | 101 | 100 | -7 | -6 | 1 | 178 | -182 | 1 | -4 | 1 | 82 | 78 |
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| 0 | 13 | 0 | 536 | -529 | -3 | -13 | 1 | 217 | -203 | -6 | -9 | 1 | 147 | -140 | -2 | -6 | 1 | 333 | 326 | 6 | -4 | 1 | 193 | 201 |
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| 4 | 13 | 0 | 118 | -120 | 2 | -13 | 1 | 160 | 180 | -3 | -9 | 1 | 70 | 66 | 1 | -6 | 1 | 387 | -352 | -7 | -3 | 1 | 236 | -243 |
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| -1 | 14 | 0 | 305 | 307 | -2 | -12 | 1 | 425 | -418 | 3 | -9 | 1 | 235 | 230 | 8 | -6 | 1 | 109 | 102 | -1 | -3 | 1 | 124 | 141 |
| 1 | 14 | 0 | 371 | -374 | -1 | -12 | 1 | 70 | 58 | 5 | -9 | 1 | 156 | -157 | -7 | -5 | 1 | 166 | 174 | 0 | -3 | 1 | 288 | 274 |
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| -1 | 15 | 0 | 110 | -115 | -4 | -11 | 1 | 109 | -94 | -2 | -8 | 1 | 189 | -173 | 0 | -5 | 1 | 163 | -129 | 6 | -3 | 1 | 86 | 91 |
| 0 | 15 | 0 | 282 | 275 | -3 | -11 | 1 | 378 | 370 | -1 | -8 | 1 | 125 | -129 | 1 | -5 | 1 | 218 | -183 | 7 | -3 | 1 | 147 | 137 |
| 2 | 15 | 0 | 320 | -315 | -2 | -11 | 1 | 187 | 190 | 0 | -8 | 1 | 50 | 42 | 2 | -5 | 1 | 332 | -332 | 8 | -3 | 1 | 127 | -123 |
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| 0 | 16 | 0 | 97 | -100 | 1 | -11 | 1 | 526 | 519 | 6 | -8 | 1 | 81 | -74 | 5 | -5 | 1 | 437 | 429 | -6 | -2 | 1 | 299 | -323 |
| 0 | -16 | 1 | 81 | 80 | 2 | -11 | 1 | 251 | -243 | -7 | -7 | 1 | 115 | -105 | 6 | -5 | 1 | 297 | 293 | -5 | -2 | 1 | 351 | 364 |
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| 3 | -16 | 1 | 116 | 130 | 4 | -11 | 1 | 54 | -52 | -4 | -7 | 1 | 475 | -468 | -8 | -4 | 1 | 123 | -121 | -3 | -2 | 1 | 342 | -323 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 7 | -1 | 1 | 139 | -134 | 4 | 2 | 1 | 257 | 258 | -2 | 5 | 1 | 361 | -371 | -4 | 8 | 1 | 72 | 76 | 3 | 11 | 1 | 262 | 262 |
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| -8 | 1 | 1 | 150 | 139 | 1 | 3 | 1 | 178 | -177 | -2 | 6 | 1 | 103 | -105 | -3 | 9 | 1 | 82 | -87 | -2 | 13 | 1 | 133 | 150 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 1 | 13 | 1 | 124 | 122 | 4 | -13 | 2 | 218 | -226 | -5 | -9 | 2 | 323 | 324 | 1 | -7 | 2 | 817 | 828 | 5 | -5 | 2 | 391 | 373 |
| 2 | 13 | 1 | 143 | -140 | 5 | -13 | 2 | 368 | 370 | -4 | -9 | 2 | 249 | -231 | 2 | -7 | 2 | 1370 | -1359 | 6 | -5 | 2 | 365 | 350 |
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| 2 | -14 | 2 | 406 | -402 | -4 | -10 | 2 | 345 | -354 | 6 | -8 | 2 | 484 | 484 | -6 | -5 | 2 | 479 | -486 | -3 | -3 | 2 | 937 | 920 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 7 | -3 | 2 | 219 | 213 | -7 | 0 | 2 | 424 | 440 | -4 | 2 | 2 | 105 | -114 | 0 | 4 | 2 | 1907 | 1938 | 5 | 6 | 2 | 297 | 301 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2 | -13 | 8 | 585 | 608 | 3 | -10 | 8 | 767 | -772 | -6 | -7 | 8 | 124 | 108 | 2 | -5 | 8 | 732 | 720 | -6 | -2 | 8 | 195 | 198 |
| 3 | -13 | 8 | 328 | -333 | 4 | -10 | 8 | 156 | 162 | -5 | -7 | 8 | 526 | -518 | 3 | -5 | 8 | 66 | -48 | -5 | -2 | 8 | 499 | 494 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -2 | 0 | 8 | 1117 | -1193 | 3 | 2 | 8 | 239 | -265 | -4 | 5 | 8 | 603 | -606 | -5 | 8 | 8 | 354 | -338 | -2 | -16 | 9 | 215 | -208 |
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| 1 | 2 | 8 | 180 | 190 | -6 | 5 | 8 | 457 | 455 | 5 | 7 | 8 | 230 | -221 | 1 | -17 | 9 | 117 | -121 | 5 | -12 | 9 | 101 | 98 |
| 2 | 2 | 8 | 1333 | 1383 | -5 | 5 | 8 | 98 | 111 | -6 | 8 | 8 | 262 | 255 | -3 | -16 | 9 | 92 | 98 | -6 | -11 | 9 | 133 | 132 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 1 | -11 | 9 | 217 | 228 | -1 | -8 | 9 | 228 | 214 | -5 | -4 | 9 | 228 | -213 | -7 | -1 | 9 | 182 | 167 | -3 | 2 | 9 | 232 | -237 |
| 2 | -11 | 9 | 186 | 190 | 0 | -8 | 9 | 149 | 145 | -4 | -4 | 9 | 114 | -109 | -6 | -1 | 9 | 143 | -139 | -2 | 2 | 9 | 81 | 95 |
| 3 | -11 | 9 | 85 | -79 | 1 | -8 | 9 | 394 | -396 | -3 | -4 | 9 | 574 | 569 | -5 | -1 | 9 | 84 | -71 | -1 | 2 | 9 | 226 | 237 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 5 | 1 | 12 | 74 | 66 | -2 | 5 | 12 | 348 | 359 | 0 | -15 | 13 | 131 | 140 | 3 | -10 | 13 | 135 | -139 | 3 | -6 | 13 | 153 | 152 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 3 | -4 | 14 | 461 | 458 | -2 | 0 | 14 | 487 | -516 | -4 | 4 | 14 | 99 | 89 | 3 | -13 | 15 | 98 | 98 | -6 | -6 | 15 | 105 | -92 |
| 4 | -4 | 14 | 86 | -79 | 0 | 0 | 14 | 638 | 650 | -3 | 4 | 14 | 207 | 213 | -2 | -12 | 15 | 208 | -214 | -5 | -6 | 15 | 99 | 98 |
| 5 | -4 | 14 | 314 | -303 | 1 | 0 | 14 | 260 | -270 | -2 | 4 | 14 | 274 | -262 | 0 | -12 | 15 | 236 | 246 | -4 | -6 | 15 | 121 | 119 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -3 | -6 | 15 | 177 | -187 | -6 | 0 | 15 | 146 | 134 | 2 | -14 | 16 | 272 | -273 | 1 | -9 | 16 | 104 | 86 | 5 | -6 | 16 | 286 | 271 |
| -1 | -6 | 15 | 261 | 274 | -3 | 0 | 15 | 80 | 70 | -2 | -13 | 16 | 250 | 250 | 2 | -9 | 16 | 479 | 463 | -6 | -5 | 16 | 128 | -134 |
| 2 | -6 | 15 | 50 | 65 | -1 | 0 | 15 | 97 | -87 | 0 | -13 | 16 | 199 | -203 | 3 | -9 | 16 | 162 | -149 | -5 | -5 | 16 | 229 | 223 |
| 3 | -6 | 15 | 95 | -109 | 0 | 0 | 15 | 158 | 172 | 1 | -13 | 16 | 181 | 169 | 4 | -9 | 16 | 299 | -287 | -3 | -5 | 16 | 470 | -458 |
| 4 | -6 | 15 | 66 | -70 | 2 | 0 | 15 | 107 | -115 | 2 | -13 | 16 | 75 | 88 | 5 | -9 | 16 | 173 | 173 | -1 | -5 | 16 | 438 | 424 |
| -6 | -5 | 15 | 139 | -139 | -5 | 1 | 15 | 178 | 174 | 3 | -13 | 16 | 282 | -278 | -5 | -8 | 16 | 100 | 121 | 0 | -5 | 16 | 275 | -269 |
| -4 | -5 | 15 | 254 | 258 | -4 | 1 | 15 | 125 | -132 | -4 | -12 | 16 | 198 | 203 | -4 | -8 | 16 | 250 | 243 | 1 | -5 | 16 | 177 | -185 |
| -2 | -5 | 15 | 84 | -81 | -2 | 1 | 15 | 154 | 153 | -3 | -12 | 16 | 244 | -243 | -3 | -8 | 16 | 176 | -174 | 2 | -5 | 16 | 544 | 533 |
| 0 | -5 | 15 | 117 | 123 | -1 | 1 | 15 | 132 | -154 | -2 | -12 | 16 | 98 | -93 | -2 | -8 | 16 | 234 | -223 | 4 | -5 | 16 | 311 | -301 |
| 1 | -5 | 15 | 226 | -234 | 3 | 1 | 15 | 64 | -68 | -1 | -12 | 16 | 372 | 358 | -1 | -8 | 16 | 502 | 489 | 5 | -5 | 16 | 185 | 181 |
| 2 | -5 | 15 | 91 | -104 | -4 | 2 | 15 | 169 | 146 | 1 | -12 | 16 | 311 | -308 | 1 | -8 | 16 | 561 | -540 | -6 | -4 | 16 | 156 | -148 |
| 4 | -5 | 15 | 74 | -97 | -1 | 2 | 15 | 70 | -66 | 2 | -12 | 16 | 147 | 151 | 2 | -8 | 16 | 125 | 102 | -5 | -4 | 16 | 142 | -151 |
| -6 | -4 | 15 | 110 | 96 | 0 | 2 | 15 | 101 | -124 | 3 | -12 | 16 | 116 | 116 | 3 | -8 | 16 | 317 | 312 | -4 | -4 | 16 | 318 | 321 |
| -5 | -4 | 15 | 183 | -183 | 1 | 2 | 15 | 168 | 158 | 4 | -12 | 16 | 196 | -207 | 4 | -8 | 16 | 292 | -277 | -2 | -4 | 16 | 405 | -409 |
| -3 | -4 | 15 | 291 | 295 | 2 | 2 | 15 | 106 | 103 | -4 | -11 | 16 | 165 | 162 | 5 | -8 | 16 | 135 | -128 | -1 | -4 | 16 | 292 | 286 |
| -2 | -4 | 15 | 90 | -106 | -5 | 3 | 15 | 86 | -58 | -3 | -11 | 16 | 210 | 200 | -5 | -7 | 16 | 232 | -239 | 0 | -4 | 16 | 423 | 412 |
| -1 | -4 | 15 | 200 | -203 | -4 | 3 | 15 | 64 | -61 | -2 | -11 | 16 | 291 | -296 | -4 | -7 | 16 | 90 | 99 | 1 | -4 | 16 | 285 | -276 |
| 0 | -4 | 15 | 67 | -71 | -1 | 3 | 15 | 77 | 87 | -1 | -11 | 16 | 220 | -207 | -3 | -7 | 16 | 285 | 272 | 2 | -4 | 16 | 201 | -192 |
| -6 | -3 | 15 | 91 | 103 | 0 | 3 | 15 | 112 | -110 | 0 | -11 | 16 | 402 | 397 | -2 | -7 | 16 | 229 | -240 | 3 | -4 | 16 | 436 | 423 |
| -4 | -3 | 15 | 242 | -234 | 2 | 3 | 15 | 165 | 156 | 2 | -11 | 16 | 322 | -320 | -1 | -7 | 16 | 285 | -268 | 4 | -4 | 16 | 180 | -171 |
| -2 | -3 | 15 | 92 | 93 | -2 | 4 | 15 | 94 | 78 | 3 | -11 | 16 | 191 | 192 | 0 | -7 | 16 | 363 | 377 | -6 | -3 | 16 | 224 | 242 |
| 0 | -3 | 15 | 142 | -156 | -1 | 4 | 15 | 85 | 90 | 4 | -11 | 16 | 172 | 165 | 1 | -7 | 16 | 131 | -136 | -5 | -3 | 16 | 193 | -195 |
| 3 | -3 | 15 | 142 | 144 | 1 | 4 | 15 | 101 | -92 | -4 | -10 | 16 | 176 | -182 | 2 | -7 | 16 | 508 | -502 | -4 | -3 | 16 | 68 | -77 |
| 5 | -3 | 15 | 91 | -93 | 1 | 5 | 15 | 58 | -62 | -3 | -10 | 16 | 146 | 148 | 3 | -7 | 16 | 140 | 143 | -3 | -3 | 16 | 371 | 371 |
| -4 | -2 | 15 | 107 | -113 | -2 | 6 | 15 | 71 | -46 | -2 | -10 | 16 | 195 | 186 | 4 | -7 | 16 | 364 | 354 | -2 | -3 | 16 | 77 | -81 |
| -3 | -2 | 15 | 126 | -134 | 0 | -16 | 16 | 173 | -179 | -1 | -10 | 16 | 448 | -443 | 5 | -7 | 16 | 160 | -166 | -1 | -3 | 16 | 314 | -318 |
| -1 | -2 | 15 | 187 | 200 | 1 | -16 | 16 | 110 | -117 | 1 | -10 | 16 | 509 | 503 | -6 | -6 | 16 | 172 | 168 | 0 | -3 | 16 | 321 | 323 |
| 0 | -2 | 15 | 103 | 105 | -2 | -15 | 16 | 282 | -280 | 2 | -10 | 16 | 74 | -71 | -5 | -6 | 16 | 69 | 74 | 1 | -3 | 16 | 155 | 166 |
| 2 | -2 | 15 | 158 | -150 | -1 | -15 | 16 | 122 | 125 | 3 | -10 | 16 | 269 | -257 | -4 | -6 | 16 | 293 | -296 | 2 | -3 | 16 | 418 | -414 |
| 3 | -2 | 15 | 67 | 77 | 0 | -15 | 16 | 199 | 209 | 4 | -10 | 16 | 258 | 242 | -2 | -6 | 16 | 279 | 282 | 4 | -3 | 16 | 250 | 242 |
| 4 | -2 | 15 | 93 | 96 | 1 | -15 | 16 | 240 | -234 | -5 | -9 | 16 | 121 | 120 | -1 | -6 | 16 | 344 | -351 | -6 | -2 | 16 | 197 | 195 |
| -5 | -1 | 15 | 72 | -76 | -3 | -14 | 16 | 122 | 112 | -4 | -9 | 16 | 105 | -109 | 0 | -6 | 16 | 315 | -302 | -5 | -2 | 16 | 220 | 212 |
| -1 | -1 | 15 | 160 | 165 | -2 | -14 | 16 | 70 | 84 | -3 | -9 | 16 | 212 | -209 | 1 | -6 | 16 | 368 | 377 | -4 | -2 | 16 | 243 | -261 |
| 0 | -1 | 15 | 116 | 140 | -1 | -14 | 16 | 239 | -241 | -2 | -9 | 16 | 305 | 300 | 2 | -6 | 16 | 93 | 74 | -3 | -2 | 16 | 117 | -107 |
| 1 | -1 | 15 | 125 | -133 | 0 | -14 | 16 | 245 | 242 | -1 | -9 | 16 | 221 | 213 | 3 | -6 | 16 | 376 | -367 | -2 | -2 | 16 | 360 | 364 |
| 2 | -1 | 15 | 122 | -126 | 1 | -14 | 16 | 181 | 191 | 0 | -9 | 16 | 512 | -497 | 4 | -6 | 16 | 224 | 217 | -1 | -2 | 16 | 159 | -169 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0 | -2 | 16 | 339 | -339 | 2 | 2 | 16 | 233 | 217 | -4 | -9 | 17 | 129 | 134 | -2 | -3 | 17 | 73 | 64 | -4 | -10 | 18 | 204 | -204 |
| 1 | -2 | 16 | 187 | 190 | -4 | 3 | 16 | 192 | 184 | -3 | -9 | 17 | 159 | -161 | 3 | -3 | 17 | 149 | 137 | -3 | -10 | 18 | 146 | 150 |
| 2 | -2 | 16 | 169 | 154 | -3 | 3 | 16 | 296 | -266 | -1 | -9 | 17 | 128 | 133 | -1 | -2 | 17 | 164 | 160 | -2 | -10 | 18 | 178 | 183 |
| 3 | -2 | 16 | 268 | -274 | -2 | 3 | 16 | 102 | -107 | 1 | -9 | 17 | 142 | -147 | 2 | -2 | 17 | 54 | -55 | -1 | -10 | 18 | 281 | -282 |
| 4 | -2 | 16 | 58 | 52 | -1 | 3 | 16 | 337 | 310 | 4 | -9 | 17 | 83 | 101 | -5 | -1 | 17 | 108 | -102 | 1 | -10 | 18 | 370 | 362 |
| -5 | -1 | 16 | 255 | 243 | 0 | 3 | 16 | 76 | -51 | -5 | -8 | 17 | 85 | -85 | -4 | -1 | 17 | 117 | 117 | 2 | -10 | 18 | 125 | -117 |
| -4 | -1 | 16 | 163 | 169 | 1 | 3 | 16 | 379 | -352 | -2 | -8 | 17 | 74 | -86 | 0 | -1 | 17 | 117 | 118 | 3 | -10 | 18 | 232 | -218 |
| -3 | -1 | 16 | 369 | -368 | 2 | 3 | 16 | 75 | 65 | 0 | -8 | 17 | 150 | 158 | 2 | -1 | 17 | 97 | -93 | -3 | -9 | 18 | 195 | -180 |
| -2 | -1 | 16 | 166 | -166 | -3 | 4 | 16 | 126 | 125 | 4 | -8 | 17 | 66 | 67 | 3 | -1 | 17 | 71 | -69 | -2 | -9 | 18 | 304 | 306 |
| -1 | -1 | 16 | 341 | 346 | -2 | 4 | 16 | 270 | -248 | -4 | -7 | 17 | 133 | -140 | -4 | 0 | 17 | 81 | -76 | -1 | -9 | 18 | 106 | 98 |
| 0 | -1 | 16 | 164 | -169 | -1 | 4 | 16 | 61 | -70 | 0 | -7 | 17 | 65 | 76 | -3 | 0 | 17 | 145 | 137 | 0 | -9 | 18 | 313 | -306 |
| 1 | -1 | 16 | 278 | -274 | 0 | 4 | 16 | 328 | 300 | 1 | -7 | 17 | 191 | 204 | 2 | 0 | 17 | 73 | -70 | 1 | -9 | 18 | 80 | 70 |
| 2 | -1 | 16 | 306 | 296 | -1 | -15 | 17 | 78 | -80 | 3 | -7 | 17 | 62 | -64 | -4 | 1 | 17 | 120 | -109 | 2 | -9 | 18 | 236 | 241 |
| 3 | -1 | 16 | 158 | 150 | 1 | -15 | 17 | 57 | 62 | -5 | -6 | 17 | 159 | 169 | -1 | 1 | 17 | 66 | -106 | 3 | -9 | 18 | 211 | -197 |
| 4 | -1 | 16 | 211 | -206 | -2 | -14 | 17 | 87 | 81 | -4 | -6 | 17 | 85 | 77 | -3 | 2 | 17 | 118 | -96 | -4 | -8 | 18 | 283 | 278 |
| -5 | 0 | 16 | 239 | -243 | 0 | -14 | 17 | 145 | -145 | -3 | -6 | 17 | 116 | -116 | -1 | 2 | 17 | 70 | 60 | -3 | -8 | 18 | 95 | -91 |
| -4 | 0 | 16 | 194 | 187 | -3 | -13 | 17 | 135 | -152 | -2 | -6 | 17 | 59 | 78 | 1 | 2 | 17 | 85 | 75 | -2 | -8 | 18 | 268 | -257 |
| -3 | 0 | 16 | 195 | 196 | -2 | -13 | 17 | 98 | -96 | -1 | -6 | 17 | 93 | 90 | -1 | -14 | 18 | 171 | -181 | -1 | -8 | 18 | 280 | 273 |
| -2 | 0 | 16 | 477 | -462 | -1 | -13 | 17 | 77 | 86 | 0 | -6 | 17 | 65 | -99 | 0 | -14 | 18 | 144 | 140 | 1 | -8 | 18 | 310 | -303 |
| -1 | 0 | 16 | 70 | -82 | 3 | -13 | 17 | 85 | 84 | 1 | -6 | 17 | 146 | 158 | 1 | -14 | 18 | 165 | 172 | 2 | -8 | 18 | 64 | 64 |
| 0 | 0 | 16 | 432 | 429 | -3 | -12 | 17 | 76 | 77 | 2 | -6 | 17 | 102 | 109 | -2 | -13 | 18 | 207 | 206 | 3 | -8 | 18 | 139 | 137 |
| 1 | 0 | 16 | 91 | -91 | -2 | -12 | 17 | 151 | -151 | 3 | -6 | 17 | 160 | -153 | 0 | -13 | 18 | 130 | -123 | -4 | -7 | 18 | 104 | 103 |
| 2 | 0 | 16 | 217 | -208 | -1 | -12 | 17 | 79 | -69 | 4 | -6 | 17 | 128 | -125 | 1 | -13 | 18 | 104 | 95 | -3 | -7 | 18 | 236 | 222 |
| 3 | 0 | 16 | 277 | 264 | 0 | -12 | 17 | 153 | 162 | -4 | -5 | 17 | 200 | 202 | 2 | -13 | 18 | 63 | 58 | -2 | -7 | 18 | 211 | -210 |
| -5 | 1 | 16 | 130 | -130 | 2 | -12 | 17 | 54 | -63 | -2 | -5 | 17 | 172 | -182 | -3 | -12 | 18 | 151 | -149 | -1 | -7 | 18 | 313 | -297 |
| -4 | 1 | 16 | 369 | -368 | -4 | -11 | 17 | 154 | -165 | -1 | -5 | 17 | 56 | 90 | -2 | -12 | 18 | 59 | -59 | 0 | -7 | 18 | 266 | 262 |
| -3 | 1 | 16 | 370 | 356 | -3 | -11 | 17 | 165 | 176 | 0 | -5 | 17 | 55 | 71 | -1 | -12 | 18 | 262 | 253 | 2 | -7 | 18 | 328 | -320 |
| -2 | 1 | 16 | 269 | 254 | -2 | -11 | 17 | 117 | 108 | 1 | -5 | 17 | 64 | -74 | 0 | -12 | 18 | 80 | -76 | 3 | -7 | 18 | 79 | 86 |
| -1 | 1 | 16 | 439 | -420 | -1 | -11 | 17 | 193 | -204 | 4 | -5 | 17 | 90 | -92 | 1 | -12 | 18 | 240 | -244 | -4 | -6 | 18 | 254 | -267 |
| 1 | 1 | 16 | 419 | 409 | 1 | -11 | 17 | 144 | 153 | -5 | -4 | 17 | 145 | -142 | 2 | -12 | 18 | 131 | 130 | -2 | -6 | 18 | 295 | 293 |
| 2 | 1 | 16 | 193 | -180 | 3 | -11 | 17 | 67 | -80 | -3 | -4 | 17 | 138 | 141 | 3 | -12 | 18 | 103 | 104 | -1 | -6 | 18 | 312 | -318 |
| 3 | 1 | 16 | 212 | -197 | -4 | -10 | 17 | 82 | -75 | -1 | -4 | 17 | 155 | -164 | -3 | -11 | 18 | 152 | 152 | 0 | -6 | 18 | 285 | -276 |
| -4 | 2 | 16 | 155 | -147 | -3 | -10 | 17 | 125 | -136 | 1 | -4 | 17 | 156 | -158 | -2 | -11 | 18 | 287 | -281 | 1 | -6 | 18 | 332 | 333 |
| -3 | 2 | 16 | 219 | -228 | -2 | -10 | 17 | 227 | 230 | 2 | -4 | 17 | 143 | 125 | 0 | -11 | 18 | 319 | 313 | 2 | -6 | 18 | 63 | 67 |
| -2 | 2 | 16 | 489 | 461 | 0 | -10 | 17 | 222 | -216 | 3 | -4 | 17 | 89 | 96 | 2 | -11 | 18 | 228 | -220 | 3 | -6 | 18 | 241 | -222 |
| 0 | 2 | 16 | 454 | -419 | 2 | -10 | 17 | 65 | 66 | -3 | -3 | 17 | 109 | -133 | 3 | -11 | 18 | 230 | 223 | -5 | -5 | 18 | 164 | 167 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -3 | -5 | 18 | 323 | -324 | -2 | -2 | 18 | 224 | 222 | -1 | -13 | 19 | 111 | 122 | -2 | -5 | 19 | 107 | -124 | -2 | -7 | 20 | 181 | -181 |
| -2 | -5 | 18 | 160 | 149 | -1 | -2 | 18 | 123 | -120 | -2 | -12 | 19 | 99 | -107 | -1 | -5 | 19 | 77 | 89 | -1 | -7 | 20 | 182 | -172 |
| -1 | -5 | 18 | 408 | 395 | 0 | -2 | 18 | 204 | -208 | 0 | -12 | 19 | 118 | 118 | -2 | -4 | 19 | 115 | -110 | 0 | -7 | 20 | 123 | 121 |
| 0 | -5 | 18 | 334 | -325 | 1 | -2 | 18 | 224 | 217 | -3 | -11 | 19 | 120 | 132 | 0 | -4 | 19 | 61 | 63 | 1 | -7 | 20 | 103 | 94 |
| 1 | -5 | 18 | 262 | -244 | 2 | -2 | 18 | 74 | 58 | -2 | -11 | 19 | 88 | 81 | -3 | -1 | 19 | 67 | 77 | 2 | -7 | 20 | 172 | -182 |
| 2 | -5 | 18 | 445 | 423 | 3 | -2 | 18 | 205 | -205 | -1 | -11 | 19 | 140 | -148 | -2 | -1 | 19 | 58 | -35 | -2 | -6 | 20 | 226 | 223 |
| -4 | -4 | 18 | 263 | 257 | -4 | -1 | 18 | 134 | 129 | -2 | -10 | 19 | 108 | 115 | 0 | -11 | 20 | 258 | 254 | -1 | -6 | 20 | 200 | -193 |
| -2 | -4 | 18 | 252 | -266 | -3 | -1 | 18 | 267 | -261 | 0 | -10 | 19 | 181 | -182 | 1 | -11 | 20 | 100 | -94 | 0 | -6 | 20 | 156 | -148 |
| -1 | -4 | 18 | 330 | 323 | -2 | -1 | 18 | 99 | -96 | -2 | -9 | 19 | 115 | -121 | -2 | -10 | 20 | 122 | 118 | 1 | -6 | 20 | 265 | 259 |
| 0 | -4 | 18 | 219 | 208 | -1 | -1 | 18 | 265 | 252 | -1 | -8 | 19 | 75 | -74 | -1 | -10 | 20 | 217 | -212 | -3 | -5 | 20 | 270 | -273 |
| 1 | -4 | 18 | 402 | -377 | 0 | -1 | 18 | 69 | -78 | 0 | -8 | 19 | 102 | 108 | 0 | -10 | 20 | 143 | 127 | -2 | -5 | 20 | 188 | 179 |
| 2 | -4 | 18 | 101 | -75 | 1 | -1 | 18 | 131 | -140 | 1 | -8 | 19 | 85 | 88 | 1 | -10 | 20 | 264 | 249 | -1 | -5 | 20 | 264 | 240 |
| 3 | -4 | 18 | 302 | 285 | 2 | -1 | 18 | 246 | 228 | -4 | -7 | 19 | 127 | -122 | -2 | -9 | 20 | 219 | 214 | 0 | -5 | 20 | 205 | -192 |
| -4 | -3 | 18 | 96 | -82 | -3 | 0 | 18 | 170 | 169 | -2 | -7 | 19 | 114 | 117 | -1 | -9 | 20 | 88 | 83 | 1 | -5 | 20 | 145 | -130 |
| -3 | -3 | 18 | 277 | 280 | -2 | 0 | 18 | 293 | -275 | 3 | -7 | 19 | 92 | -93 | 0 | -9 | 20 | 153 | -153 | -2 | -4 | 20 | 197 | -190 |
| -2 | -3 | 18 | 90 | -92 | 0 | 0 | 18 | 344 | 320 | -3 | -6 | 19 | 99 | -94 | 1 | -9 | 20 | 72 | 75 | -1 | -4 | 20 | 202 | 204 |
| -1 | -3 | 18 | 227 | -227 | 1 | 0 | 18 | 79 | -86 | -2 | -6 | 19 | 143 | 145 | 2 | -9 | 20 | 118 | 104 | 0 | -4 | 20 | 152 | 141 |
| 0 | -3 | 18 | 286 | 276 | -3 | 1 | 18 | 285 | 266 | -1 | -6 | 19 | 85 | 85 | -2 | -8 | 20 | 151 | -135 | 1 | -4 | 20 | 283 | -266 |
| 1 | -3 | 18 | 65 | 76 | -2 | 1 | 18 | 109 | 115 | 0 | -6 | 19 | 125 | -120 | -1 | -8 | 20 | 180 | 179 | -2 | -3 | 20 | 77 | -71 |
| 2 | -3 | 18 | 365 | -351 | -1 | 1 | 18 | 306 | -288 | 2 | -6 | 19 | 58 | 61 | 1 | -8 | 20 | 246 | -244 | -1 | -3 | 20 | 192 | -187 |
| -4 | -2 | 18 | 106 | -105 | 1 | 1 | 18 | 201 | 197 | -4 | -5 | 19 | 149 | 148 | 2 | -8 | 20 | 85 | 79 | 0 | -3 | 20 | 140 | 131 |
| -3 | -2 | 18 | 122 | -119 | -2 | 2 | 18 | 241 | 228 | -3 | -5 | 19 | 86 | -93 | -3 | -7 | 20 | 195 | 190 | | | | | |

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR p-Ethoxyphenyl[2-(2-pyridyl)phenyl]telluride 

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2 | 0 | 0 | 426 | -482 | 3 | 3 | 0 | 295 | 318 | 2 | 6 | 0 | 247 | -227 | 6 | 9 | 0 | 700 | -685 | 1 | 14 | 0 | 327 | -349 |
| 3 | 0 | 0 | 144 | -149 | 5 | 3 | 0 | 230 | -253 | 3 | 6 | 0 | 998 | -1022 | 7 | 9 | 0 | 619 | -644 | 5 | 14 | 0 | 111 | 125 |
| 4 | 0 | 0 | 1243 | -1364 | 6 | 3 | 0 | 264 | -296 | 4 | 6 | 0 | 934 | -961 | 8 | 9 | 0 | 171 | -175 | 6 | 14 | 0 | 117 | 125 |
| 5 | 0 | 0 | 1002 | -1094 | 7 | 3 | 0 | 178 | -194 | 5 | 6 | 0 | 586 | -623 | 9 | 9 | 0 | 153 | 144 | 9 | 14 | 0 | 158 | -149 |
| 6 | 0 | 0 | 665 | -686 | 8 | 3 | 0 | 128 | -128 | 6 | 6 | 0 | 185 | -193 | 10 | 9 | 0 | 430 | 406 | 1 | 15 | 0 | 299 | 310 |
| 7 | 0 | 0 | 188 | 213 | 9 | 3 | 0 | 193 | 207 | 7 | 6 | 0 | 391 | 395 | 11 | 9 | 0 | 219 | 212 | 2 | 15 | 0 | 425 | 439 |
| 8 | 0 | 0 | 746 | 798 | 10 | 3 | 0 | 401 | 409 | 8 | 6 | 0 | 528 | 533 | 14 | 9 | 0 | 183 | -182 | 3 | 15 | 0 | 279 | 290 |
| 9 | 0 | 0 | 623 | 665 | 11 | 3 | 0 | 138 | 143 | 9 | 6 | 0 | 370 | 383 | 0 | 10 | 0 | 448 | 428 | 5 | 15 | 0 | 214 | -207 |
| 10 | 0 | 0 | 282 | 290 | 12 | 3 | 0 | 118 | 112 | 10 | 6 | 0 | 256 | 273 | 2 | 10 | 0 | 202 | -193 | 6 | 15 | 0 | 300 | -291 |
| 11 | 0 | 0 | 368 | -374 | 14 | 3 | 0 | 112 | -103 | 12 | 6 | 0 | 324 | -323 | 4 | 10 | 0 | 103 | -99 | 7 | 15 | 0 | 244 | -255 |
| 12 | 0 | 0 | 498 | -493 | 0 | 4 | 0 | 470 | 490 | 13 | 6 | 0 | 191 | -180 | 5 | 10 | 0 | 184 | -166 | 9 | 15 | 0 | 154 | 157 |
| 13 | 0 | 0 | 258 | -242 | 1 | 4 | 0 | 1514 | 1553 | 1 | 7 | 0 | 846 | 884 | 8 | 10 | 0 | 124 | 121 | 10 | 15 | 0 | 188 | 186 |
| 14 | 0 | 0 | 163 | -161 | 3 | 4 | 0 | 990 | -1007 | 2 | 7 | 0 | 766 | 784 | 11 | 10 | 0 | 133 | -141 | 0 | 16 | 0 | 299 | -313 |
| 2 | 1 | 0 | 450 | 472 | 4 | 4 | 0 | 914 | -962 | 3 | 7 | 0 | 740 | 782 | 1 | 11 | 0 | 495 | 534 | 1 | 16 | 0 | 155 | -171 |
| 3 | 1 | 0 | 107 | 107 | 5 | 4 | 0 | 923 | -977 | 5 | 7 | 0 | 533 | -561 | 2 | 11 | 0 | 715 | 725 | 3 | 16 | 0 | 173 | 171 |
| 4 | 1 | 0 | 90 | -88 | 6 | 4 | 0 | 129 | -128 | 6 | 7 | 0 | 691 | -712 | 3 | 11 | 0 | 561 | 587 | 4 | 16 | 0 | 162 | 153 |
| 6 | 1 | 0 | 92 | -112 | 7 | 4 | 0 | 598 | 619 | 7 | 7 | 0 | 653 | -670 | 4 | 11 | 0 | 170 | 163 | 5 | 16 | 0 | 147 | 145 |
| 7 | 1 | 0 | 286 | -299 | 8 | 4 | 0 | 662 | 680 | 9 | 7 | 0 | 290 | 298 | 5 | 11 | 0 | 384 | -404 | 8 | 16 | 0 | 135 | -134 |
| 8 | 1 | 0 | 101 | -77 | 9 | 4 | 0 | 477 | 507 | 10 | 7 | 0 | 373 | 380 | 6 | 11 | 0 | 619 | -617 | 9 | 16 | 0 | 193 | -184 |
| 11 | 1 | 0 | 93 | 83 | 10 | 4 | 0 | 175 | 181 | 11 | 7 | 0 | 414 | 408 | 7 | 11 | 0 | 438 | -442 | 2 | 17 | 0 | 254 | 247 |
| 0 | 2 | 0 | 1631 | 1686 | 12 | 4 | 0 | 306 | -308 | 12 | 7 | 0 | 101 | 115 | 9 | 11 | 0 | 272 | 281 | 3 | 17 | 0 | 202 | 195 |
| 1 | 2 | 0 | 824 | 854 | 13 | 4 | 0 | 271 | -255 | 14 | 7 | 0 | 147 | -126 | 10 | 11 | 0 | 363 | 360 | 5 | 17 | 0 | 152 | -156 |
| 2 | 2 | 0 | 687 | -723 | 1 | 5 | 0 | 757 | 760 | 0 | 8 | 0 | 1055 | 1053 | 11 | 11 | 0 | 241 | 246 | 6 | 17 | 0 | 225 | -211 |
| 3 | 2 | 0 | 1111 | -1151 | 2 | 5 | 0 | 755 | 797 | 1 | 8 | 0 | 428 | 425 | 8 | 12 | 0 | 87 | -56 | 7 | 17 | 0 | 168 | -168 |
| 4 | 2 | 0 | 1035 | -1130 | 3 | 5 | 0 | 660 | 673 | 3 | 8 | 0 | 132 | -155 | 1 | 13 | 0 | 498 | 504 | 0 | 18 | 0 | 277 | -283 |
| 5 | 2 | 0 | 849 | -916 | 4 | 5 | 0 | 427 | 430 | 4 | 8 | 0 | 157 | -164 | 2 | 13 | 0 | 668 | 678 | 1 | 18 | 0 | 105 | -112 |
| 7 | 2 | 0 | 464 | 500 | 5 | 5 | 0 | 376 | -383 | 5 | 8 | 0 | 209 | -221 | 3 | 13 | 0 | 481 | 489 | 4 | 18 | 0 | 225 | 225 |
| 8 | 2 | 0 | 744 | 809 | 6 | 5 | 0 | 756 | -800 | 7 | 8 | 0 | 192 | 191 | 4 | 13 | 0 | 91 | 96 | 5 | 18 | 0 | 169 | 162 |
| 9 | 2 | 0 | 721 | 749 | 7 | 5 | 0 | 479 | -500 | 8 | 8 | 0 | 243 | 249 | 5 | 13 | 0 | 295 | -299 | 2 | 19 | 0 | 149 | 157 |
| 10 | 2 | 0 | 106 | 129 | 8 | 5 | 0 | 211 | -218 | 9 | 8 | 0 | 136 | 138 | 6 | 13 | 0 | 462 | -447 | -15 | 1 | 1 | 219 | 201 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 6 | 9 | 1 | 203 | -208 | -6 | 12 | 1 | 459 | 479 | 2 | 14 | 1 | 269 | 279 | 1 | 17 | 1 | 301 | -299 | 5 | 0 | 2 | 1041 | -1072 |
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| -13 | 10 | 1 | 216 | 208 | -1 | 12 | 1 | 740 | -763 | 7 | 14 | 1 | 292 | -281 | 6 | 17 | 1 | 186 | 177 | 9 | 0 | 2 | 118 | 139 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 6 | 1 | 5 | 369 | 362 | 5 | 3 | 5 | 544 | 547 | 1 | 5 | 5 | 644 | -621 | -2 | 7 | 5 | 472 | 463 | 2 | 9 | 5 | 203 | -211 |
| 7 | 1 | 5 | 306 | 317 | 6 | 3 | 5 | 572 | 570 | 2 | 5 | 5 | 612 | -603 | -1 | 7 | 5 | 194 | 196 | 3 | 9 | 5 | 139 | -144 |
| 8 | 1 | 5 | 185 | -179 | 7 | 3 | 5 | 217 | 216 | 3 | 5 | 5 | 113 | -97 | 0 | 7 | 5 | 225 | -216 | 5 | 9 | 5 | 196 | 155 |
| 9 | 1 | 5 | 408 | -404 | 9 | 3 | 5 | 314 | -306 | 4 | 5 | 5 | 354 | 352 | 1 | 7 | 5 | 379 | -364 | 6 | 9 | 5 | 131 | 141 |
| 10 | 1 | 5 | 337 | -329 | 10 | 3 | 5 | 367 | -379 | 5 | 5 | 5 | 450 | 447 | 2 | 7 | 5 | 454 | -438 | 7 | 9 | 5 | 104 | 116 |
| 11 | 1 | 5 | 187 | -190 | -13 | 4 | 5 | 125 | -136 | 6 | 5 | 5 | 334 | 342 | 4 | 7 | 5 | 134 | 137 | -10 | 10 | 5 | 172 | 183 |
| 13 | 1 | 5 | 173 | 165 | -10 | 4 | 5 | 112 | 116 | 7 | 5 | 5 | 174 | 169 | 5 | 7 | 5 | 223 | 225 | -9 | 10 | 5 | 255 | 249 |
| -9 | 2 | 5 | 156 | 146 | -9 | 4 | 5 | 208 | 207 | 9 | 5 | 5 | 277 | -275 | 6 | 7 | 5 | 206 | 200 | -8 | 10 | 5 | 236 | 244 |
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| 3 | 2 | 5 | 166 | -163 | 1 | 4 | 5 | 100 | 40 | -2 | 6 | 5 | 366 | 358 | -5 | 8 | 5 | 396 | -401 | 3 | 10 | 5 | 529 | -524 |
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| 9 | 2 | 5 | 196 | 204 | 5 | 4 | 5 | 196 | -187 | 2 | 6 | 5 | 243 | -249 | -1 | 8 | 5 | 700 | 667 | 7 | 10 | 5 | 327 | 327 |
| -12 | 3 | 5 | 237 | 245 | 6 | 4 | 5 | 295 | 290 | 3 | 6 | 5 | 551 | -532 | 0 | 8 | 5 | 583 | 537 | 8 | 10 | 5 | 310 | 315 |
| -11 | 3 | 5 | 298 | 298 | 7 | 4 | 5 | 408 | 397 | 4 | 6 | 5 | 488 | -470 | 1 | 8 | 5 | 146 | 125 | 9 | 10 | 5 | 151 | 143 |
| -10 | 3 | 5 | 188 | 195 | 8 | 4 | 5 | 177 | 189 | 5 | 6 | 5 | 95 | -91 | 2 | 8 | 5 | 298 | -308 | -4 | 11 | 5 | 159 | -159 |
| -8 | 3 | 5 | 349 | -351 | 9 | 4 | 5 | 154 | 156 | 7 | 6 | 5 | 284 | 265 | 3 | 8 | 5 | 546 | -547 | -9 | 12 | 5 | 207 | 207 |
| -7 | 3 | 5 | 640 | -656 | 11 | 4 | 5 | 138 | -159 | 8 | 6 | 5 | 312 | 316 | 4 | 8 | 5 | 395 | -388 | -8 | 12 | 5 | 202 | 219 |
| -6 | 3 | 5 | 547 | -551 | 12 | 4 | 5 | 133 | -122 | 11 | 6 | 5 | 159 | -148 | 5 | 8 | 5 | 148 | -134 | -6 | 12 | 5 | 284 | -264 |
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| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 4 | 12 | 5 | 370 | -361 | 10 | 0 | 6 | 221 | -230 | 1 | 3 | 6 | 242 | 228 | -1 | 5 | 6 | 194 | 184 | 0 | 7 | 6 | 506 | 470 |
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| -3 | 13 | 5 | 133 | -129 | 3 | 1 | 6 | 118 | -129 | 6 | 3 | 6 | 192 | -182 | 5 | 5 | 6 | 309 | -314 | 7 | 7 | 6 | 149 | 154 |
| -6 | 14 | 5 | 125 | -126 | 4 | 1 | 6 | 186 | -179 | 8 | 3 | 6 | 113 | 123 | 8 | 5 | 6 | 186 | 174 | 8 | 7 | 6 | 255 | 259 |
| -5 | 14 | 5 | 322 | -316 | 7 | 1 | 6 | 111 | 98 | -11 | 4 | 6 | 177 | 167 | 9 | 5 | 6 | 158 | 168 | 9 | 7 | 6 | 241 | 239 |
| -4 | 14 | 5 | 193 | -191 | -11 | 2 | 6 | 230 | 233 | -10 | 4 | 6 | 255 | 258 | 10 | 5 | 6 | 118 | 121 | -10 | 8 | 6 | 110 | 134 |
| -1 | 14 | 5 | 304 | 301 | -10 | 2 | 6 | 307 | 333 | -9 | 4 | 6 | 193 | 209 | -10 | 6 | 6 | 125 | 140 | -6 | 8 | 6 | 208 | -205 |
| 0 | 14 | 5 | 353 | 337 | -9 | 2 | 6 | 227 | 237 | -7 | 4 | 6 | 240 | -246 | -9 | 6 | 6 | 115 | 141 | -5 | 8 | 6 | 303 | -300 |
| 2 | 14 | 5 | 123 | -155 | -7 | 2 | 6 | 196 | -198 | -6 | 4 | 6 | 431 | -431 | -8 | 6 | 6 | 126 | 90 | -4 | 8 | 6 | 168 | -163 |
| 3 | 14 | 5 | 293 | -307 | -6 | 2 | 6 | 469 | -456 | -5 | 4 | 6 | 346 | -334 | -7 | 6 | 6 | 175 | -209 | -2 | 8 | 6 | 187 | 175 |
| 4 | 14 | 5 | 265 | -259 | -5 | 2 | 6 | 588 | -590 | -3 | 4 | 6 | 301 | 293 | -6 | 6 | 6 | 349 | -356 | -1 | 8 | 6 | 269 | 263 |
| -3 | 15 | 5 | 112 | -104 | -4 | 2 | 6 | 157 | -178 | -2 | 4 | 6 | 693 | 679 | -5 | 6 | 6 | 190 | -165 | 2 | 8 | 6 | 167 | -181 |
| 5 | 15 | 5 | 126 | -93 | -3 | 2 | 6 | 379 | 375 | -1 | 4 | 6 | 695 | 698 | -3 | 6 | 6 | 200 | 179 | 3 | 8 | 6 | 97 | -107 |
| -1 | 16 | 5 | 201 | 226 | -2 | 2 | 6 | 648 | 628 | 0 | 4 | 6 | 194 | 185 | -2 | 6 | 6 | 384 | 390 | 4 | 8 | 6 | 140 | -158 |
| 0 | 16 | 5 | 215 | 216 | -1 | 2 | 6 | 622 | 603 | 1 | 4 | 6 | 307 | -305 | -1 | 6 | 6 | 470 | 445 | 6 | 8 | 6 | 172 | 185 |
| -11 | 0 | 6 | 219 | 221 | 0 | 2 | 6 | 164 | 167 | 2 | 4 | 6 | 429 | -431 | 1 | 6 | 6 | 197 | -200 | 7 | 8 | 6 | 136 | 141 |
| -10 | 0 | 6 | 318 | 337 | 1 | 2 | 6 | 477 | -454 | 3 | 4 | 6 | 544 | -564 | 2 | 6 | 6 | 423 | -407 | -9 | 9 | 6 | 257 | 253 |
| -9 | 0 | 6 | 201 | 197 | 2 | 2 | 6 | 562 | -547 | 4 | 4 | 6 | 126 | -118 | 3 | 6 | 6 | 370 | -374 | -8 | 9 | 6 | 336 | 350 |
| -7 | 0 | 6 | 342 | -347 | 3 | 2 | 6 | 364 | -368 | 6 | 4 | 6 | 115 | 118 | 4 | 6 | 6 | 96 | -71 | -7 | 9 | 6 | 154 | 115 |
| -6 | 0 | 6 | 608 | -617 | 4 | 2 | 6 | 217 | -217 | 7 | 4 | 6 | 360 | 376 | 5 | 6 | 6 | 110 | 113 | -5 | 9 | 6 | 337 | -333 |
| -5 | 0 | 6 | 504 | -501 | 5 | 2 | 6 | 138 | 121 | 8 | 4 | 6 | 120 | 119 | 6 | 6 | 6 | 261 | 272 | -4 | 9 | 6 | 503 | -460 |
| -4 | 0 | 6 | 94 | -121 | 6 | 2 | 6 | 479 | 475 | 9 | 4 | 6 | 131 | -127 | 7 | 6 | 6 | 295 | 295 | -3 | 9 | 6 | 354 | -344 |
| -3 | 0 | 6 | 419 | 400 | 7 | 2 | 6 | 520 | 506 | 10 | 4 | 6 | 228 | -227 | 8 | 6 | 6 | 135 | 127 | -2 | 9 | 6 | 167 | -164 |
| -2 | 0 | 6 | 791 | 795 | 8 | 2 | 6 | 183 | 188 | 11 | 4 | 6 | 197 | -201 | 9 | 6 | 6 | 95 | -66 | -1 | 9 | 6 | 238 | 242 |
| -1 | 0 | 6 | 556 | 531 | 9 | 2 | 6 | 92 | -97 | -11 | 5 | 6 | 140 | -104 | 10 | 6 | 6 | 158 | -154 | 0 | 9 | 6 | 481 | 461 |
| 1 | 0 | 6 | 494 | -484 | 10 | 2 | 6 | 191 | -201 | -9 | 5 | 6 | 193 | 200 | -9 | 7 | 6 | 241 | 248 | 1 | 9 | 6 | 401 | 425 |
| 2 | 0 | 6 | 751 | -733 | 11 | 2 | 6 | 202 | -200 | -8 | 5 | 6 | 223 | 241 | -8 | 7 | 6 | 285 | 295 | 2 | 9 | 6 | 184 | 181 |
| 3 | 0 | 6 | 314 | -304 | -8 | 3 | 6 | 228 | 245 | -7 | 5 | 6 | 222 | 218 | -7 | 7 | 6 | 204 | 215 | 3 | 9 | 6 | 199 | -209 |
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| 5 | 0 | 6 | 144 | 149 | -4 | 3 | 6 | 305 | -294 | -5 | 5 | 6 | 223 | -227 | -5 | 7 | 6 | 294 | -280 | 5 | 9 | 6 | 291 | -276 |
| 6 | 0 | 6 | 775 | 774 | -2 | 3 | 6 | 113 | 106 | -4 | 5 | 6 | 435 | -427 | -4 | 7 | 6 | 263 | -238 | 6 | 9 | 6 | 162 | -168 |
| 7 | 0 | 6 | 343 | 354 | -1 | 3 | 6 | 207 | 191 | -3 | 5 | 6 | 454 | -469 | -3 | 7 | 6 | 302 | -288 | 8 | 9 | 6 | 217 | 221 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 9 | 9 | 6 | 202 | 198 | -2 | 1 | 7 | 448 | 453 | -2 | 4 | 7 | 160 | -153 | 0 | 7 | 7 | 173 | 158 | -5 | 0 | 8 | 139 | -127 |
| -5 | 10 | 6 | 154 | -153 | -1 | 1 | 7 | 645 | 635 | 1 | 4 | 7 | 301 | 290 | 3 | 7 | 7 | 249 | -267 | -4 | 0 | 8 | 493 | -543 |
| -4 | 10 | 6 | 201 | -197 | 0 | 1 | 7 | 265 | 264 | 2 | 4 | 7 | 147 | 155 | 4 | 7 | 7 | 259 | -259 | -3 | 0 | 8 | 293 | -294 |
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| 4 | 10 | 6 | 137 | -116 | 3 | 1 | 7 | 541 | -549 | -9 | 5 | 7 | 214 | 218 | 8 | 7 | 7 | 149 | 150 | 1 | 0 | 8 | 387 | 388 |
| 6 | 10 | 6 | 125 | 131 | 4 | 1 | 7 | 481 | -493 | -8 | 5 | 7 | 200 | 225 | -8 | 8 | 7 | 181 | 179 | 2 | 0 | 8 | 108 | 148 |
| -8 | 11 | 6 | 255 | 279 | 5 | 1 | 7 | 312 | -303 | -6 | 5 | 7 | 245 | -264 | -7 | 8 | 7 | 276 | 299 | 3 | 0 | 8 | 164 | -149 |
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| -4 | 11 | 6 | 281 | -286 | 8 | 1 | 7 | 213 | 217 | -1 | 5 | 7 | 304 | 301 | -3 | 8 | 7 | 363 | -367 | 6 | 0 | 8 | 162 | -172 |
| -3 | 11 | 6 | 349 | -348 | -4 | 2 | 7 | 204 | -205 | 0 | 5 | 7 | 392 | 407 | -2 | 8 | 7 | 327 | -339 | -2 | 1 | 8 | 173 | -149 |
| -2 | 11 | 6 | 118 | -65 | -3 | 2 | 7 | 190 | -203 | 1 | 5 | 7 | 145 | 154 | 0 | 8 | 7 | 211 | 207 | -1 | 1 | 8 | 127 | -127 |
| -1 | 11 | 6 | 260 | 274 | 1 | 2 | 7 | 306 | 318 | 2 | 5 | 7 | 113 | -115 | 1 | 8 | 7 | 301 | 294 | -7 | 2 | 8 | 233 | 271 |
| 0 | 11 | 6 | 414 | 400 | 5 | 2 | 7 | 155 | -145 | 3 | 5 | 7 | 253 | -247 | 2 | 8 | 7 | 362 | 362 | -5 | 2 | 8 | 246 | -258 |
| 1 | 11 | 6 | 355 | 353 | 6 | 2 | 7 | 219 | -224 | 4 | 5 | 7 | 302 | -311 | 3 | 8 | 7 | 161 | 155 | -4 | 2 | 8 | 296 | -324 |
| 2 | 11 | 6 | 100 | 103 | -10 | 3 | 7 | 144 | 146 | 7 | 5 | 7 | 207 | 220 | 4 | 8 | 7 | 121 | -157 | -3 | 2 | 8 | 264 | -272 |
| 3 | 11 | 6 | 140 | -153 | -9 | 3 | 7 | 207 | 230 | 8 | 5 | 7 | 248 | 234 | 5 | 8 | 7 | 275 | -269 | -1 | 2 | 8 | 133 | 123 |
| 4 | 11 | 6 | 323 | -325 | -8 | 3 | 7 | 187 | 215 | -8 | 6 | 7 | 157 | 151 | 6 | 8 | 7 | 241 | -232 | 0 | 2 | 8 | 364 | 375 |
| 5 | 11 | 6 | 309 | -308 | -6 | 3 | 7 | 235 | -247 | -7 | 6 | 7 | 221 | 226 | -1 | 9 | 7 | 128 | 125 | 1 | 2 | 8 | 410 | 417 |
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| -6 | 1 | 7 | 178 | -173 | -8 | 4 | 7 | 141 | 145 | -4 | 7 | 7 | 297 | -279 | 1 | 12 | 7 | 280 | 312 | 0 | 4 | 8 | 274 | 276 |
| -5 | 1 | 7 | 506 | -514 | -7 | 4 | 7 | 139 | 160 | -2 | 7 | 7 | 140 | 150 | 2 | 12 | 7 | 254 | 266 | 1 | 4 | 8 | 351 | 351 |
| -4 | 1 | 7 | 487 | -485 | -3 | 4 | 7 | 287 | -295 | -1 | 7 | 7 | 246 | 264 | -7 | 0 | 8 | 143 | 148 | 2 | 4 | 8 | 127 | 129 |

| H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC | H | K | L | 10FO | 10FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 | 4 | 8 | 150 | -158 | 2 | 5 | 8 | 256 | 280 | 1 | 6 | 8 | 185 | 207 | 1 | 7 | 8 | 114 | 112 | 1 | 9 | 8 | 142 | 146 |
| 4 | 4 | 8 | 320 | -333 | 3 | 5 | 8 | 177 | 190 | 4 | 6 | 8 | 127 | -157 | 2 | 7 | 8 | 198 | 213 | 2 | 9 | 8 | 260 | 255 |
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| -1 | 5 | 8 | 123 | -145 | 0 | 6 | 8 | 184 | 217 | 0 | 7 | 8 | 146 | -121 | -1 | 9 | 8 | 264 | -277 | | | | | |

# APPENDIX E

# SUPPORTING PUBLICATIONS

## List of Supporting Publications

(1) "Bis(2-(2-pyridyl)phenyl)tritelluride - Synthesis and Crystal Structure". Thomas A Hamor, Najih Al-Salim, Antony A West and William R McWhinnie. Journal of Organometallic Chemistry, 310 (1986) C5-C7.

(2) "2-Pyridyl- and Quinolin-2-yl-functionalised Organyl -tellurium Ligands. The stabilisation of Diorganyl Tritellurides. The Crystal and Molecular Structures of 2-(2-Pyridyl)phenyltellurium(IV) Tribromide, Dimethyl dithiocarbamato [2-(2-pyridyl) phenyl]tellurium(II) and *p*-Ethoxyphenyl-2-(2-Pyridyl)phenyl Telluride". Najih Al-Salim, Antony A West, William R McWhinnie and Thomas A Hamor. J. Chem. Soc. Dalton Transactions, 1988, 2363-2371.

(3) "The Crystal and Molecular Structure of Dimethyldithiocarbamato-2- (2-quinolinyl)phenyl-tellurium(II)". Antony A West, William R McWhinnie and Thomas A Hamor. Journal of Organometallic Chemistry, 356, (1988), 159-164.

(4) "The Synthesis and Reactions of 1,6-Bis-2-butyltelluro -phenyl-2-5-diazahexa-1,5-diene and Related Compounds. The Crystal and Molecular Structures of 2-dichloro(butyl)tellurobenzaldehyde and Bis-[2-(N-hydroxy)iminophenyl] Ditelluride". Khalid Y Abid, Najih Al-Salim, Martin Greaves, William R McWhinnie, Antony A West and Thomas A Hamor. Accepted for publication by J. Chem. Soc. Dalton Transactions.

(5) "*Ortho*-Tellurated Derivatives of N,N-Dimethylbenzyl-amine: Crystal and Molecular Structures of (2-N,N-Dimethylbenzylamine-C,N')tellurium(IV) Tribromide and 2-Dichlorobutotelluro-N-dimethylbenzylammonium chloride". Harkesh B. Singh, Narsimhan Sudha, Antony A West and Thomas A Hamor Accepted for publiction by J. Chem. Soc. Dalton Transactions.

(6) "The Lewis Acidity of Organylbismuth(III) Compounds. New Organylbismuth (III) Dithiocarbamate Chemistry. The Crystal and Molecular Structure of Bis-(diethyldithiocarbamato)phenyl bismuth(III) and of Bis-(diethyldithiocarbamato)[2-(2-pyridyl)phenyl]-bismuth(III)". Mushtaq Ali, William R McWhinnie, Antony A West and Thomas A Hamor. Accepted for publication by J. Chem. Soc. Dalton Transactions.

*Journal of Organometallic Chemistry*, 310 (1986) C5–C7
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands

**Preliminary communication**

# BIS(2-(2-PYRIDYL)PHENYL)TRITELLURIDE – SYNTHESIS AND CRYSTAL STRUCTURE

THOMAS A. HAMOR

*Department of Chemistry, University of Birmingham, Birmingham B15 2TT (Great Britain)*

NAJIH AL-SALIM, ANTONY A. WEST, and WILLIAM R. MCWHINNIE

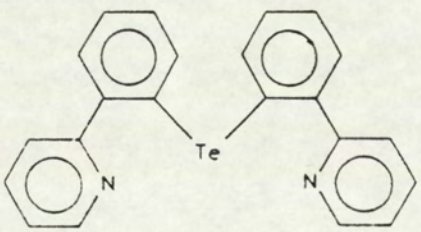
*(Department of Molecular Sciences, Aston University, Aston Triangle, Birmingham B4 7ET (Great Britain)*

(Received April 18th, 1986)

## Summary

The synthesis and molecular structure of bis(2-(2-pyridyl)phenyl)tritelluride are described. There is a short Te · · · N non-bonded interaction of 2.554(7) Å, which may be responsible for the stability of the molecule. The tritelluride can be converted into the related ditelluride by treatment with copper in refluxing dioxane. The ditelluride may also be prepared by a route not involving the tritelluride.

Whilst attempting to prepare the functionalised tellurium-containing ligand (I), we tried, by use of $NaBH_4$, to reduce an organyltellurium tribromide (III) directly to the sodium organyltelluride( − 1). Instead a novel tritelluride was isolated in good yield (see Scheme 1). The tritelluride was converted into the ditelluride (V) by treatment with copper powder in refluxing dioxane.

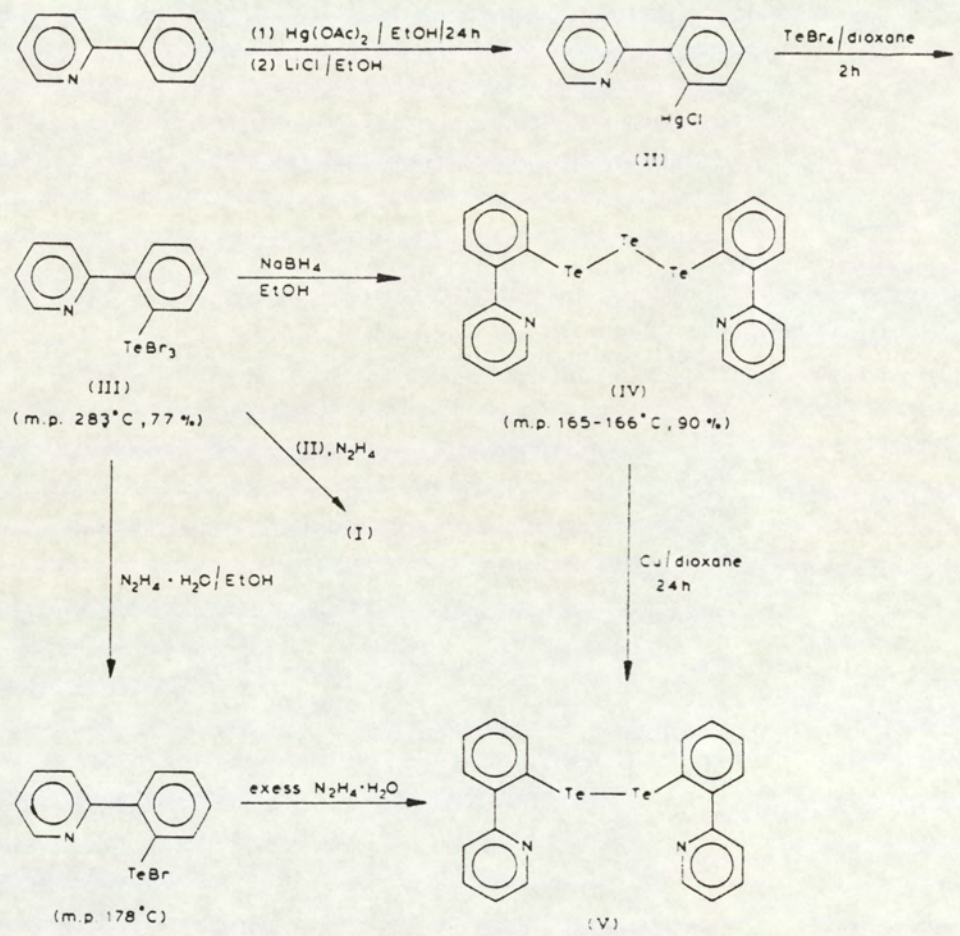



(I)

Crystals of IV suitable for single crystal X-ray diffraction studies were obtained from benzene/toluene:

*Crystal data.* $C_{22}H_{16}N_2Te_3$. $M_r = 691.2$, monoclinic, space group $I2/c$, $a$ 14.721(3), $b$ 9.290(4), $c$ 15.996(10) Å, $\beta$ 106.39(3)°, $U$ 2098.7 Å$^3$, $Z = 4$, $D_c$ 2.188 g cm$^{-3}$, $F(000) = 1272$.

0022-328X/86/$03.50 




SCHEME 1

Cell dimensions and intensities were measured with an Enraf–Nonius CAD-4 diffractometer using monochromated Mo-$K_\alpha$ radiation. 1634 reflections in the range $2 < \theta < 25°$ having $I > 2.5\sigma(I)$ were used in the analysis. The structure was solved by Patterson and Fourier methods and refined by least-squares with SHELX [1], using anisotropic temperature factors for all non-hydrogen atoms. Hydrogen atoms were located from a difference map and included in fixed positions. The calculations were terminated when all shifts were $< 0.1$ and $R$ and $R_w$ were 0.064 and 0.088, respectively; $w = 1/(\sigma^2(F) + 0.005F^2)$.

A stereoscopic view of the structure of IV drawn with PLUTO [2] is shown in Fig. 1. The compound exhibits two-fold symmetry with the central tellurium atom lying on a crystallographic two fold axis *.

* The atomic coordinates for this structure are available on request from the Director of the Cambridge Crystallographic Data Centre, University Chemical laboratory, Lensfield Road, Cambridge CB2 1EW (Great Britain). Any request should be accompanied by the full literature citation for this communication.

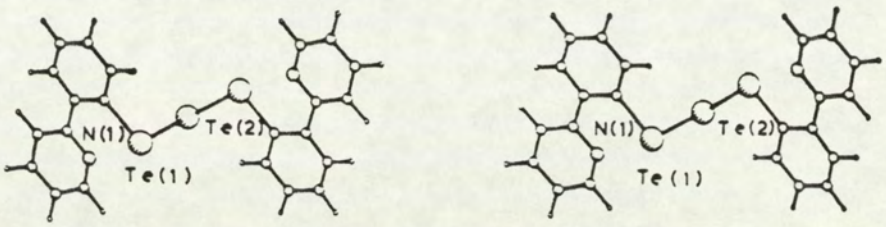



Fig. 1. Stereoscopic view of the molecule. Selected bond lengths (Å) and angles (°): Te(1)–Te(2), 2.776(1); Te(1)–C, 2.130(9); Te(1) · · · N, 2.554(7); Te(1)–Te(2)–Te(1)', 100.4(1); C–Te(1)–Te(2), 99.1(2); N · · · Te(1)–Te(2), 170.7(2); N · · · Te(1)–C, 71.6(3).

Of particular note is the short Te · · · N non-bonded interaction of 2.554(7) Å, which appears to hold the ligand in an essentially planar geometry; the pyridyl and phenyl rings are, nevertheless, bent very slightly relative to one another to form a shallow V shape, with internal angle 177.2(4)°. The coordination of Te(1), by Te(2), N, and its bonded carbon atom is accurately planar. The torsion angle about the Te(1)–Te(2) bond is 97.3(3)°.

During the course of this work the structure of bis(tris(trimethylsilyl)methyl)tritelluride was reported [3]. The Te–Te bond length found in that compound (2.710(1) Å) is shorter by 0.066 Å than the corresponding length in our structure. This difference may be a manifestation of somewhat stronger Te–C binding involving an aromatic carbon atom (Te–C shorter by 0.083 Å) in our compound.

Apart from the tritelluride ion, $[Te_3]^{2-}$ [4], $(Me_3Si)_3C_2Te_3$ and the present compound are the only tritellurides reported to date. The bulky $(Me_3Si)_3C$ groups are believed to contribute to the stability of the former compound [3]. The diorganyltritelluride reported here, by contrast, appears to owe its stability to the significant Te · · · N interaction, suggesting that if the organic ligands have a suitably positioned donor atom, many more successful syntheses of tritellurides should be possible.

**Acknowledgement.** One of us (N. Al-S.) thanks the Government of Iraq for a scholarship.

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# 2-Pyridyl- and Quinolin-2-yl-functionalised Organyltellurium Ligands. The Stabilisation of Diorganyl Tritellurides. The Crystal and Molecular Structures of 2-(2-Pyridyl)phenyltellurium(IV) Tribromide, Dimethyldithiocarbamato[2-(2-pyridyl)phenyl]tellurium(II), and *p*-Ethoxyphenyl 2-(2-Pyridyl)phenyl Telluride*

**Najih Al-Salim, Antony A. West, and William R. McWhinnie**
*Department of Chemical Engineering and Applied Chemistry, Aston University, Aston Triangle, Birmingham B4 7ET*
**Thomas A. Hamor**
*Department of Chemistry, University of Birmingham, P.O. Box 363, Birmingham B15 2TT*

The syntheses and spectroscopic data ($^{13}C$ n.m.r.) for some new organyltellurium ligands are described. Thus *trans* metallation of 2-(2-pyridyl)phenyl- (R) or 2-(quinolin-2-yl)phenyl- (R') mercury(II) chloride with $TeBr_4$ gives the organyltellurium tribromides $RTeBr_3$ and $R'TeBr_3$, which may be reduced to the organyltellurium bromides RTeBr and R'TeBr, some metathesis reactions of which are described; or, in the presence of excess hydrazine, to diorganyl ditellurides, $R_2Te_2$ and $R'_2Te_2$. Reduction of $RTeBr_3X$ with $NaBH_4$ gave a novel tritelluride, $R_2Te_3$; a method of preparation for $R'_2Te_3$ was also devised. Syntheses of the tellurides $R_2Te$, $R'_2Te$, and $RTe(C_6H_4OEt\text{-}p)$ were developed and the intermediacy of ionic compounds $[R_2TeBr][HgClBr_2]$ and $[R_2TeCl][HgCl_3]$ noted. The crystal structures of 2-(2-pyridyl)phenyltellurium(IV) tribromide, (**1**), dimethyldithiocarbamato[2-(2-pyridyl)phenyl]tellurium(II), (**2**), and *p*-ethoxyphenyl 2-(2-pyridyl)phenyltelluride, (**3**), have been determined from three-dimensional *X*-ray counter data. Complex (**1**) is triclinic, space group $P\bar{1}$ with $a = 6.953(6)$, $b = 8.382(1)$, $c = 12.133(2)$ Å, $\alpha = 78.68(1)$, $\beta = 82.87(4)$, $\gamma = 87.14(4)°$, and $Z = 2$; $R = 0.0735$ for 1 963 observed reflections. The co-ordination about Te is octahedral with a vacant equatorial position, two Br atoms apical, the third Br and the organic group equatorial [Te–C, 2.110(19); Te–N, 2.244(14) Å]. The compound is essentially monomeric, with a weak association between Te and Br [3.596(3) Å] in a neighbouring molecule. Complex (**2**) is triclinic, space group $P\bar{1}$ with $a = 8.809(3)$, $b = 9.032(5)$, $c = 10.727(4)$ Å, $\alpha = 83.06(4)$, $\beta = 86.49(3)$, $\gamma = 63.68(4)$, and $Z = 2$; $R = 0.0417$ for 2 502 observed reflections. The co-ordination about Te is trigonal bipyramidal, C(1) and two lone pairs of Te comprising the equatorial co-ordination and the S(1) atom together with the pyridyl-N atom axial [Te–C, 2.111(5); Te–N, 2.354(4) Å]. No significant intermolecular contacts occur. Complex (**3**) is monoclinic, space group $P2_1/c$ with $a = 13.422(2)$, $b = 16.469(3)$, $c = 7.711(3)$ Å, $\beta = 91.01(2)°$, and $Z = 4$; $R = 0.0415$ for 2 014 observed reflections. The Te atom is bonded to two C atoms. The N atom of the pyridyl ring is twisted away from Te [Te–N, 2.695(4) Å] by rotation of the pyridyl ring about the pyridyl–phenyl bond by 23.2(3)°, a manifestation of the *trans* effect of the Te–C(ethoxyphenyl) covalent bond.

Current objectives include the synthesis of ligands which are bidentate or multidentate containing only tellurium atoms[1] and also the preparation of ligands which, in addition to tellurium, contain a harder donor atom, *e.g.* oxygen or nitrogen. We have reported briefly on our synthesis of organyltellurides functionalised with imino groups[2] and in this paper describe the syntheses of 2-pyridyl- and quinolin-2-yl-functionalised organyltellurium compounds. These materials constitute a new class of ligand, although a few related organylselenium compounds and their complexes are known, *e.g.* bis[2-(2-pyridyl)ethyl] selenide (L),[3] a terdentate ligand which forms complexes $[Cu(L)X_2]$ (X = Cl, Br, $NO_3$, or $ClO_4$). The preparation of 2-(2-pyridyl)benzo[*b*]selenophene (L') has been reported together with the palladium(II) complex, $[Pd(L')Cl_2]$.[4] Amongst known selenium ligands containing a quinoline nucleus may be listed 8-methylselenoquinoline[5] and quinoline-8-selenol.[6]

During the course of the preparative work novel tritellurides were isolated, a preliminary account of which has appeared;[7] full preparative details are given here. Following that earlier structural study[7] of bis[2-(2-pyridyl)phenyl] tritelluride in which a short Te–N [2.554(7) Å] interaction occurs, we now also report the crystal structures of 2-(2-pyridyl)phenyltellurium(IV) tribomide, (**1**), dimethyldithiocarbamato[2-(2-pyridyl)phenyl]tellurium(II), (**2**), and *p*-ethoxyphenyl 2-(2-pyridyl)phenyl telluride, (**3**). We examine the effect of tellurium oxidation state and ligand type *trans* to nitrogen on the Te–N interaction.

## Experimental

*Synthesis of New Organyltellurium Compounds.*—(a) *2-Pyridyl derivatives. 2-(2-Pyridyl)phenylmercury(II) chloride.* 2-Phenylpyridine (7.81 g, 50 mmol) and mercury(II) acetate (15.93 g, 50 mmol) were mixed in absolute ethanol (100 $cm^3$) and refluxed for 24 h, after which LiCl (4.4 g) in absolute ethanol (100 $cm^3$) was added with vigorous stirring; the thick mixture was heated

* *Supplementary data available*: see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1988, Issue 1, pp. xvii–xx.

for a further 15 min. The reaction mixture was poured into distilled water (300 $cm^3$) and filtered. The filtrate was left to stand at room temperature whereupon a white crystalline compound formed which was filtered off, washed with cold methanol, and oven dried at 60 °C; yield 35.5%, m.p. 138—140 °C.

2-(2-*Pyridyl*)*phenyltellurium*(IV) *tribomide*, (**1**). A solution of 2-(2-pyridyl)phenylmercury(II) chloride (3.9 g, 10 mmol) in dry 1,4-dioxane (20 $cm^3$) was added to tellurium tetrabromide (4.47 g, 10 mmol) dissolved in dry 1,4-dioxane (15 $cm^3$). The mixture was refluxed under dry dinitrogen for 2 h during which time a yellow compound precipitated. The hot mixture was filtered and the yellow product washed with pure ethanol and recrystallised from acetonitrile–methanol (1:1) to give yellow crystals; yield 77%, m.p. 283—285 °C.

2-(2-*Pyridyl*)*phenyltellurium*(II)*bromide*. 2-(2-Pyridyl)phenyltellurium(IV) tribromide (1.04 g, 2 mmol) was suspended in absolute ethanol. Hydrazine hydrate (0.1 g, 2 mmol) was added to the stirred mixture under dinitrogen. The solid dissolved at the completion of the addition and the solution was refluxed for 15 min and set aside to cool to room temperature. Yellow crystals deposited which were filtered off and washed with ethanol; yield 65%, m.p. 178—180 °C.

*Bis*[2-(2-*pyridyl*)*phenyl*] *ditelluride*. 2-(2-Pyridyl)phenyltellurium(IV) tribomide (1.04 g, 2 mmol) was dissolved in acetonitrile–ethanol (2:1) (30 $cm^3$) and treated with excess hydrazine hydrate (0.6 g, 12 mmol) in ethanol (10 $cm^3$) and then refluxed for 20 min. The precipitate so formed was filtered off and recrystallised from benzene to give yellow-orange crystals; yield 60%, m.p. 150—152 °C.

*Bis*[2-(2-*pyridyl*)*phenyl*] *tritelluride*. 2-(2-Pyridyl)phenyltellurium(IV) tribomide (2.6 g, 5 mmol) was suspended in absolute ethanol (60 $cm^3$) and treated dropwise under dinitrogen with a solution of sodium tetrahydroborate (1.1 g, 30 mmol) in ethanol. The solid dissolved as the addition proceeded, then a black precipitate of tellurium formed; this then redissolved to give, finally, a red solution. The solution was heated for 10 min, filtered hot and allowed to cool to room temperature. A red-brown solid appeared which was filtered off and recrystallised from benzene–toluene; yield 90% (based on Te content of the tribromide), m.p. 165—166 °C.

*Reaction of bis*[2-(2-*pyridyl*)*phenyl*] *tritelluride with copper metal*. Bis[2-(2-pyridyl)phenyl] tritelluride (1.38 g, 2 mmol) dissolved in 1,4-dioxane (15 $cm^3$) was treated with copper powder (0.38 g, 6 mmol). The mixture was refluxed for 24 h and then filtered. Removal of the solvent *in vacuo* gave a residue which, on recrystallisation from ethanol gave golden yellow crystals of bis[2-(2-pyridyl)phenyl] ditelluride (by analysis, m.p., and mixed m.p.).

*Bis*[2-(2-*pyridyl*)*phenyl*] *telluride*. 2-(2-Pyridyl)phenyltellurium(IV) tribromide (1.04 g, 2 mmol) and 2-(2-pyridyl)-phenylmercury(II) chloride (0.78 g, 2 mmol) were dissolved in dry 1,4-dioxane (20 $cm^3$) and refluxed for 3 h. The white solid thus formed was filtered off and washed with dry methanol, followed by recrystallisation from nitromethane, m.p. 235—238 °C. Analysis indicated the composition $C_{22}H_{16}Br_3Cl$-$HgN_2Te$ (Found: C, 29.3; H, 1.80; N, 3.10. $C_{22}H_{16}Br_3ClHgN_2Te$ requires C, 29.0; H, 1.75; N, 3.05%). The white solid (0.91 g, 1 mmol) was suspended in ethanol, stirred, and treated dropwise with a solution of hydrazine hydrate (0.2 g, 5 mmol) in ethanol (5 $cm^3$) after which it was heated. Elemental mercury deposited and the solution became yellow. After filtering, water was added to the filtrate to precipitate a yellow compound which was filtered off, washed with water, and recrystallised from ethanol; yield 65%, m.p. 102—103 °C.

*Bis*[2-(2-*pyridinio*)*phenyl*]*tellurium*(II)*diperchlorate*. Bis[2-(2-pyridyl)phenyl] telluride (0.44 g, 1 mmol) was dissolved in hot methanol (30 $cm^3$) and treated with an excess of perchloric acid. The solution was heated with stirring for 30 min and filtered. The filtrate was set aside and, over a period of time, a yellow crystalline compound formed which was filtered off, washed with ethanol, and air dried; m.p. 270 °C (decomp.).

p-*Ethoxyphenyl* 2-(2-*pyridyl*)*phenyl telluride*, (**3**). 2-(2-Pyridyl)phenylmercury(II) chloride (1.95 g, 5 mmol) and *p*-ethoxyphenyltellurium(IV) trichloride[8] (1.28 g, 5 mmol) were dissolved in dry 1,4-dioxane (20 $cm^3$) and refluxed for 2 h. The white product was filtered from the hot solution, washed with methanol, and oven dried at 100 °C (Found: C, 30.4; H, 2.50; N, 1.60. $C_{19}H_{17}Cl_4NOTe$ requires C, 30.6; H, 2.30; N, 1.85%). The white compound was suspended in ethanol (30 $cm^3$) and treated dropwise with an excess of hydrazine hydrate and stirred for 15 min. Water (50 $cm^3$) was added and the reaction mixture filtered. The solid obtained was recrystallised from ethanol–acetone (1:1) to give a pale yellow crystalline compound; yield 67%, m.p. 124—126 °C.

*Dimethyldithiocarbamato*[2-(2-*pyridyl*)*phenyl*]*tellurium*(II) (**2**). 2-(2-Pyridyl)phenyltellurium(II) bromide (0.36 g, 1 mmol) in acetonitrile (15 $cm^3$) was treated with an acetonitrile solution (15 $cm^3$) of sodium dimethyldithiocarbamate [Na(dmdtc)] (0.18 g, 1 mmol); the mixture was then heated for 30 min. The solution was filtered and the filtrate left to cool to afford a yellow crystalline material; 75% yield, m.p. 165—167 °C.

(b) *Quinolin-2-yl derivatives*. 2-(*Quinolin-2-yl*)*phenylmercury*(II) *chloride*. 2-Phenylquinoline[9] (m.p. 84—85 °C, lit.,[9] 85—86 °C) (10.25 g, 50 mmol) and mercury(II) acetate (15.93 g, 50 mmol) were mixed and refluxed in absolute ethanol (100 $cm^3$). After 7 h, LiCl (4.4 g) in methanol (30 $cm^3$) was added after which the procedure was identical to that for the 2-pyridyl derivative. The white product had m.p. 180—182 °C and yield 34%.

2-(*Quinolin-2-yl*)*phenyltellurium*(IV) *tribromide*, 2-(*quinolin-2-yl*)*phenyltellurium*(II) *bromide*, *and bis*[2-(*quinolin-2-yl*)-*phenyl*] *ditelluride*. These compounds (m.p. 340, 150, and 220—222 °C respectively) were all prepared by methods analogous to those developed for the 2-(2-pyridyl)phenyl derivatives.

*Bis*[2-(*quinolin-2-yl*)*phenyl*] *telluride*. Bis[2-(quinolin-2-yl)-phenyl] ditelluride (1.33 g, 2 mmol) and copper powder (0.38 g, 6 mmol) were stirred under reflux in 1,4-dioxane (15 $cm^3$) for 24 h. The mixture was filtered and the solvent removed *in vacuo*. The residue was recrystallised from ethanol to give a yellow crystalline compound; yield 70%, m.p. 187—189 °C.

*Bis*[2-(*quinolin-2-yl*)*phenyl*] *tritelluride*. Sodium tetrahydroborate reduction of 2-(quinolin-2-yl)phenyltellurium(IV) tribromide gave only the ditelluride; also no precipitated tellurium was seen during the reaction. The following method was therefore adopted: 2-(quinolin-2-yl)phenyltellurium(IV) tribromide (2.28 g, 4 mmol) was suspended in ethanol (25 $cm^3$), tellurium powder (0.26 g, 4 mmol) was then added and the mixture stirred under argon. Sodium tetrahydroborate (1.1 g) in ethanol (20 $cm^3$) was then introduced slowly until all the tellurium had dissolved, the resulting red solution was refluxed for 30 min and then set aside to cool. An orange product formed which was filtered and recrystallised from benzene to give an 80% yield of an orange-brown compound, m.p. 211—212 °C.

*Dimethyldithiocarbamato*[2-(*quinolin-2-yl*)*phenyl*]*tellurium*-(II). This compound, m.p. 206 °C, was prepared by the method used for the 2-pyridyl analogue.

Analytical data for the compounds are given in Table 1. The Scheme summarises the syntheses and interconversions within the 2-(2-pyridyl)phenyl series.

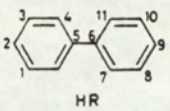
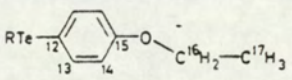
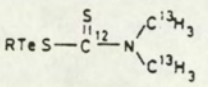
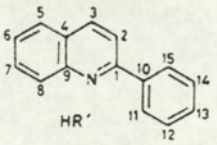
*Physical Measurements*.—Melting points (uncorrected) were determined with a Gallenkamp electrically heated apparatus. The molar conductance ($10^{-3}$ mol $dm^{-3}$ solutions) was measured at room temperature with a Mullard bridge and dip cell with bright platinum electrodes (type E 591 B).

**Table 1.** Analytical data for new organyltellurium compounds; R = 2-(2-pyridyl)phenyl, R′ = 2-(quinolin-2-yl)phenyl, dmdtc = dimethyldithiocarbamate

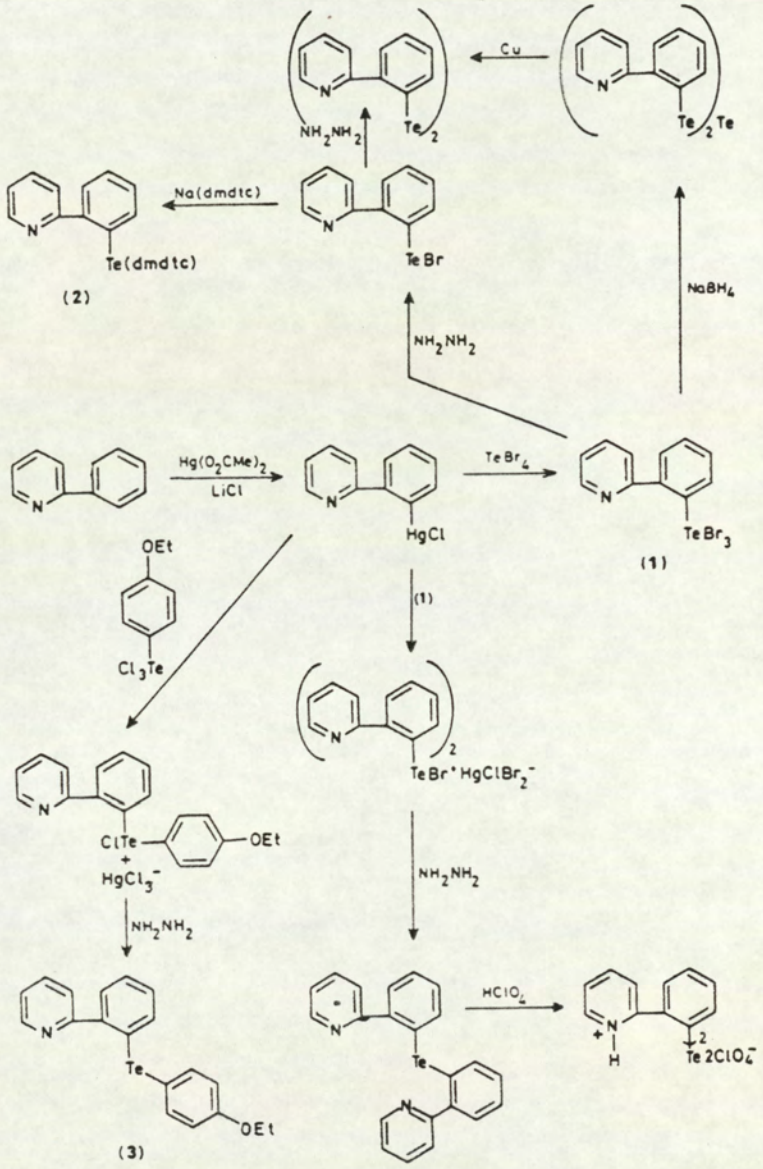
| Compound | Colour | Found (%) | | | | | Calc. (%) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | C | H | N | Br | Te | C | H | N | Br | Te |
| RHgCl | White | 33.7 | 2.00 | 3.50 | | | 33.8 | 2.10 | 3.60 | | |
| $RTeBr_3$ | Yellow | 25.7 | 1.40 | 2.80 | 46.3 | | 25.3 | 1.60 | 2.70 | 46.0 | |
| RTeBr | Yellow | 37.0 | 2.20 | 3.80 | 22.3 | | 36.5 | 2.20 | 3.85 | 22.1 | |
| $R_2Te_2$ | Yellow-orange | 46.6 | 3.00 | 5.20 | | 45.4 | 46.9 | 2.90 | 5.00 | | 46.0 |
| $R_2Te_3$ | Brown-red | 39.0 | 2.30 | 3.90 | | 55.6 | 38.2 | 2.30 | 4.00 | | 55.4 |
| $R_2Te$ | Yellow | 60.5 | 3.70 | 6.30 | | 28.5 | 60.6 | 3.70 | 6.40 | | 29.2 |
| $[(HR)_2Te][ClO_4]$[a] | Dull yellow | 41.6 | 2.60 | 4.10 | | | 41.5 | 2.50 | 4.40 | | |
| $R(C_6H_4OEt\text{-}p)$ | Pale yellow | 56.5 | 4.10 | 3.40 | | 31.1 | 56.6 | 4.30 | 3.45 | | 31.7 |
| RTe(dmdtc) | Yellow | 42.0 | 3.50 | 7.00 | < 1 | | 41.8 | 3.50 | 6.95 | 0.0 | |
| HR′ | White | 87.2 | 5.20 | 6.80 | | | 87.8 | 5.40 | 6.80 | | |
| R′HgCl | White | 40.9 | 2.10 | 2.90 | | | 40.9 | 2.30 | 3.20 | | |
| $R'TeBr_3$ | Bright yellow | 31.3 | 1.70 | 2.60 | 41.9 | | 31.2 | 1.75 | 2.45 | 41.9 | |
| R′TeBr | Yellow | 43.9 | 2.30 | 3.20 | 19.3 | | 43.8 | 2.45 | 3.40 | 19.4 | |
| $R'_2Te_2$ | Orange | 54.1 | 2.90 | 3.90 | | 38.1 | 54.3 | 3.04 | 4.20 | | 38.5 |
| $R'_2Te$ | Yellow | 67.5 | 3.50 | 4.90 | | 23.6 | 67.2 | 3.75 | 5.20 | | 23.8 |
| $R'_2Te_3$ | Orange-brown | 45.8 | 2.30 | 3.50 | | 47.5 | 45.5 | 2.55 | 3.55 | | 48.4 |
| R′Te(dmdtc) | Yellow | 48.0 | 3.30 | 6.40 | | | 47.8 | 3.55 | 6.20 | | |
| $[R_2TeBr][HgClX_2]$[b] | White | 29.3 | 1.80 | 3.10 | | | 29.0 | 1.75 | 3.05 | | |
| $[R_2TeCl][HgCl_3]$[c] | White | 30.4 | 2.50 | 1.60 | | | 30.6 | 2.30 | 1.85 | | |

[a] $\Lambda(CH_3CN)$, 251 $ohm^{-1}\ cm^2\ mol^{-1}$. [b] $\Lambda(CH_3NO_2)$, 73.7 $ohm^{-1}\ cm^2\ mol^{-1}$. [c] $\Lambda(Me_2SO)$, 33.6 $ohm^{-1}\ cm^2\ mol^{-1}$.

**Table 2.** Some $^{13}C$ n.m.r. data for organylmercury and organyltellurium derivatives of 2-phenylpyridine (HR) and 2-phenylquinoline (HR′)

| Compound (solvent) | $^{13}C$ Chemical shifts (p.p.m.) relative to $SiMe_4$ | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $C^1$ | $C^2$ | $C^3$ | $C^4$ | $C^5$ | $C^6$ | $C^7$ | $C^8$ | $C^9$ | $C^{10}$ | $C^{11}$ | |
|  HR (CDCl₃) | 149.1 | 119.9 | 136.2 | 121.5 | 156.7 | 138.8<br>139.6[a] | 126.3<br>127.1[a] | 128.7[a] | 128.1<br>129[a] | 128.7[a] | 126.3<br>127.1[a] | |
| RHgCl (Me₂SO) | 149.5 | 120.9 | 138.3 | 123.4 | 155.9 | 141.3<br>136.5[b] | 147.9<br>151[b] | 137.8<br>136.5[b] | 128.2<br>127.9[b] | 129.2<br>127.6[b] | 127.1<br>127.9[b] | |
| $RTeBr_3$ (Me₂SO) | 145.1 | 122.7 | 133.6 | 126.4 | 151.5 | | 144.3 | 132.2 | 129.3 | 131.1 | 127.9 | |
|  (CDCl₃) | | 145.7<br>109.7<br>($C^{12}$) | 119.9<br>141.3<br>($C^{13}$) | 134.4<br>115.7<br>($C^{14}$) | 121.7<br>159.7<br>($C^{15}$) | 142.1<br>63.2<br>($C^{16}$) | 115.3<br>14.7<br>($C^{17}$) | 136.8 | 128.9 | 132.2 | 127 | |
|  (CDCl₃) | 142.9 | 119.9 | 134.6 | 122.2 | 154.1 | 134.9 | 130.1 | 137.9 | 126.3 | 130.3 | 126.1 | 44.7<br>($C^{13}$) |
| $R_2Te_3$ (CDCl₃) | 146 | 119 | 137.2 | 122.3 | | 140.2 | 120.4 | 140.7 | 129.5 | 125.6 | 126.8 | |
|  HR′ (Me₂SO) | 156.2<br>128.9<br>($C^{12}$) | 118.8<br>129.1<br>($C^{13}$) | 137.2 | 127.8 | 127.2 | 126.5 | 129.6 | 129.9 | 147.6 | 138.7 | 127 | |
| R′HgCl (Me₂SO) | 156<br>130.0<br>($C^{13}$) | 119.1<br>128.2<br>($C^{14}$) | 135<br>127.4<br>($C^{15}$) | 138.2<br>($C^{12}$) | 127.9 | 126.8 | | 130.4 | 146.3 | 141.1 | 150 | |
| $R'TeBr_3$ (Me₂SO) | 150.9<br>131.3<br>($C^{13}$) | 118.9 | 132.7<br>127.8<br>($C^{15}$) | 134.4<br>($C^{12}$) | 128.5 | 124.5 | 129.6 | 131.8 | 144.7 | 140.8 | | |

[a] Data for 2,4,6-triphenylpyridine. [b] Data for phenylmercury(II) chloride.[18]




**Scheme.** Synthesis and interconversions of 2-pyridyl-functionalised organyltellurium compounds

I.r. spectra were determined for KBr discs with a Perkin-Elmer 599B instrument. $^1H$ And $^{13}C$ n.m.r. spectra were obtained with a JEOL FX 90Q spectrometer at 90 and 22.5 MHz respectively; $SiMe_4$ was the internal standard in both cases. Analysis for tellurium was by a literature method,[10] other analyses were provided by the Analytical Services Unit, Department of Molecular Sciences, Aston University.

$^{13}C$ N.m.r. data are given in Table 2.

*Structural Investigations.*—Crystal samples of (1), (2), and (3) were subjected to preliminary study by photographic methods, but the final cell dimensions and reflection intensities were measured with graphite-monochromated Mo-$K_\alpha$ radiation on an Enraf–Nonius CAD-4 diffractometer, operating in the $\omega$–$2\theta$ scan mode. Three standard reflections were monitored at regular intervals to check the stability of the system. Lorentz and polarisation factors were applied, and also empirical absorption corrections in the case of compound (1);[11] range of transmission factors 0.524—1.083. Details of crystal and experimental parameters are given in Table 3.

The tellurium and bromine atoms of (1) were located by direct methods. For (2) and (3) the tellurium atom was located

Table 3. Crystal and experimental parameters

| Complex | (1) | (2) | (3) |
|---|---|---|---|
| Molecular formula | $C_{11}H_8Br_3NTe$ | $C_{14}H_{14}N_2S_2Te$ | $C_{19}H_{17}NOTe$ |
| $M$ | 521.5 | 402.0 | 402.9 |
| Crystal size (mm) | 0.4 × 0.2 × 0.1 | 0.6 × 0.25 × 0.075 | 0.55 × 0.25 × 0.125 |
| $a$/Å | 6.953(6) | 8.809(3) | 13.422(2) |
| $b$/Å | 8.382(1) | 9.032(5) | 16.469(3) |
| $c$/Å | 12.133(2) | 10.727(4) | 7.711(3) |
| $\alpha$/° | 78.68(1) | 83.06(4) | 90.00 |
| $\beta$/° | 82.87(4) | 86.49(3) | 91.01(2) |
| $\gamma$/° | 87.14(4) | 63.68(4) | 90.00 |
| $U$/Å$^3$ | 687.8 | 759.4 | 1 704.2 |
| Space group | $P\bar{1}$ | $P\bar{1}$ | $P2_1/c$ |
| $Z$ | 2 | 2 | 4 |
| $F(000)$ | 476 | 392 | 792 |
| $D_c$/g cm$^{-3}$ | 2.519 | 1.759 | 1.571 |
| $\mu$(Mo-$K_\alpha$)/mm$^{-1}$ | 11.453 | 2.282 | 1.813 |
| Data collection range, $\theta$/° ($\lambda$ = 0.710 69 Å) | 2—25 | 2—25 | 2—22.5 |
| Unique data measured | 2 697 | 2 940 | 3 356 |
| Significant data [$I > 2.5\sigma(I)$] | 1 963 | 2 502 | 2 014 |
| Least-squares weight, $K$ {$w = [\sigma^2(F) + KF^2]^{-1}$} | 0 | 0.0025 | 0.0015 |
| Maximum shift/error in final least-squares cycle | 0.15 | 0.01 | 0.01 |
| Final $R$ | 0.0735 | 0.0417 | 0.0415 |
| Final $R'$ | 0.1085 | 0.0636 | 0.0539 |

Table 4. Bond lengths (Å) and angles (°) for compound (1) with e.s.d.s in parentheses

| | | | |
|---|---|---|---|
| Te–Br(1) | 2.673(3) | C(5)–C(6) | 1.410(25) |
| Te–Br(2) | 2.658(3) | C(6)–C(1) | 1.438(25) |
| Te–Br(3) | 2.589(3) | C(6)–C(7) | 1.415(28) |
| Te–C(1) | 2.110(19) | C(8)–C(9) | 1.435(34) |
| Te–N(1) | 2.244(14) | C(8)–C(7) | 1.441(25) |
| C(2)–C(3) | 1.312(29) | C(9)–C(10) | 1.312(35) |
| C(2)–C(1) | 1.373(29) | C(10)–C(11) | 1.397(28) |
| C(3)–C(4) | 1.471(29) | C(11)–N(1) | 1.295(25) |
| C(4)–C(5) | 1.357(30) | N(1)–C(7) | 1.407(21) |
| | | | |
| Br(1)–Te–Br(2) | 172.4(1) | C(5)–C(6)–C(7) | 121.7(16) |
| Br(1)–Te–Br(3) | 93.5(2) | C(1)–C(6)–C(7) | 121.2(17) |
| Br(2)–Te–Br(3) | 92.8(1) | Te–C(1)–C(2) | 125.4(16) |
| Br(1)–Te–C(1) | 87.5(5) | Te–C(1)–C(6) | 112.8(14) |
| Br(2)–Te–C(1) | 88.1(5) | C(2)–C(1)–C(6) | 121.7(18) |
| Br(3)–Te–C(1) | 92.5(6) | C(9)–C(8)–C(7) | 117.4(20) |
| Br(1)–Te–N(1) | 88.4(4) | C(8)–C(9)–C(10) | 120.2(21) |
| Br(2)–Te–N(1) | 84.7(4) | C(9)–C(10)–C(11) | 119.2(23) |
| Br(3)–Te–N(1) | 170.4(4) | C(10)–C(11)–N(1) | 123.4(22) |
| C(1)–Te–N(1) | 78.2(7) | Te–N(1)–C(11) | 126.8(14) |
| C(3)–C(2)–C(1) | 118.9(21) | Te–N(1)–C(7) | 113.0(11) |
| C(2)–C(3)–C(4) | 122.9(21) | C(11)–N(1)–C(7) | 120.3(17) |
| C(3)–C(4)–C(5) | 117.0(20) | C(6)–C(7)–C(8) | 127.1(17) |
| C(4)–C(5)–C(6) | 121.5(19) | C(6)–C(7)–N(1) | 114.7(15) |
| C(5)–C(6)–C(1) | 117.0(17) | C(8)–C(7)–N(1) | 117.8(18) |

Table 5. Bond lengths (Å) and angles (°) for compound (2) with e.s.d.s in parentheses

| | | | |
|---|---|---|---|
| Te–S(1) | 2.518(1) | C(1)–C(6) | 1.406(7) |
| Te–N(1) | 2.354(4) | C(2)–C(3) | 1.375(8) |
| Te–C(1) | 2.111(5) | C(3)–C(4) | 1.367(9) |
| S(1)–C(12) | 1.764(4) | C(4)–C(5) | 1.398(8) |
| S(2)–C(12) | 1.676(5) | C(5)–C(6) | 1.373(7) |
| N(1)–C(7) | 1.332(7) | C(6)–C(7) | 1.464(6) |
| N(1)–C(11) | 1.333(7) | C(7)–C(8) | 1.410(7) |
| N(2)–C(12) | 1.319(6) | C(8)–C(9) | 1.353(9) |
| N(2)–C(13) | 1.478(7) | C(9)–C(10) | 1.395(10) |
| N(2)–C(14) | 1.464(7) | C(10)–C(11) | 1.372(8) |
| C(1)–C(2) | 1.400(6) | | |
| | | | |
| S(1)–Te–N(1) | 167.6(1) | C(3)–C(4)–C(5) | 120.7(5) |
| S(1)–Te–C(1) | 95.0(1) | C(4)–C(5)–C(6) | 119.7(5) |
| N(1)–Te–C(1) | 74.4(2) | C(1)–C(6)–C(5) | 119.5(5) |
| Te–S(1)–C(12) | 109.6(1) | C(1)–C(6)–C(7) | 118.1(4) |
| Te–N(1)–C(7) | 113.8(3) | C(5)–C(6)–C(7) | 122.4(5) |
| Te–N(1)–C(11) | 125.2(4) | N(1)–C(7)–C(6) | 115.8(4) |
| C(7)–N(1)–C(11) | 120.9(5) | N(1)–C(7)–C(8) | 119.8(5) |
| C(12)–N(2)–C(13) | 121.8(5) | C(6)–C(7)–C(8) | 124.4(5) |
| C(12)–N(2)–C(14) | 123.4(5) | C(7)–C(8)–C(9) | 119.0(5) |
| C(13)–N(2)–C(14) | 114.8(4) | C(8)–C(9)–C(10) | 120.5(5) |
| Te–C(1)–C(2) | 122.0(4) | C(9)–C(10)–C(11) | 117.7(5) |
| Te–C(1)–C(6) | 118.0(3) | N(1)–C(11)–C(10) | 122.0(5) |
| C(2)–C(1)–C(6) | 120.1(4) | S(1)–C(12)–S(2) | 122.3(3) |
| C(1)–C(2)–C(3) | 119.3(5) | S(1)–C(12)–N(2) | 115.1(4) |
| C(2)–C(3)–C(4) | 120.7(5) | S(2)–C(12)–N(2) | 122.7(4) |

Table 6. Bond lengths (Å) and angles (°) for compound (3) with e.s.d.s in parentheses

| | | | |
|---|---|---|---|
| Te–C(1) | 2.138(6) | C(6)–C(7) | 1.468(9) |
| Te–C(12) | 2.144(6) | C(7)–C(8) | 1.394(10) |
| O(1)–C(15) | 1.368(8) | C(8)–C(9) | 1.359(11) |
| O(1)–C(18) | 1.430(9) | C(9)–C(10) | 1.364(12) |
| N(1)–C(7) | 1.325(8) | C(10)–C(11) | 1.384(11) |
| N(1)–C(11) | 1.331(9) | C(12)–C(13) | 1.372(10) |
| C(1)–C(2) | 1.387(9) | C(12)–C(17) | 1.393(10) |
| C(1)–C(6) | 1.415(9) | C(13)–C(14) | 1.382(10) |
| C(2)–C(3) | 1.380(10) | C(14)–C(15) | 1.380(10) |
| C(3)–C(4) | 1.382(11) | C(15)–C(16) | 1.372(10) |
| C(4)–C(5) | 1.346(10) | C(16)–C(17) | 1.408(10) |
| C(5)–C(6) | 1.410(9) | C(18)–C(19) | 1.463(12) |
| | | | |
| C(1)–Te–C(12) | 94.8(2) | C(7)–C(8)–C(9) | 119.5(8) |
| C(15)–O(1)–C(18) | 118.1(6) | C(8)–C(9)–C(10) | 120.2(8) |
| C(7)–N(1)–C(11) | 118.9(7) | C(9)–C(10)–C(11) | 117.4(7) |
| Te–C(1)–C(2) | 120.2(5) | N(1)–C(11)–C(10) | 123.2(8) |
| Te–C(1)–C(6) | 119.8(4) | Te–C(12)–C(13) | 119.8(5) |
| C(2)–C(1)–C(6) | 120.0(6) | Te–C(12)–C(17) | 121.9(5) |
| C(1)–C(2)–C(3) | 120.6(7) | C(13)–C(12)–C(17) | 118.3(6) |
| C(2)–C(3)–C(4) | 119.4(7) | C(12)–C(13)–C(14) | 122.2(7) |
| C(3)–C(4)–C(5) | 121.1(7) | C(13)–C(14)–C(15) | 119.2(7) |
| C(4)–C(5)–C(6) | 121.5(7) | O(1)–C(15)–C(14) | 116.2(7) |
| C(1)–C(6)–C(5) | 117.3(6) | O(1)–C(15)–C(16) | 123.3(7) |
| C(1)–C(6)–C(7) | 122.2(6) | C(14)–C(15)–C(16) | 120.5(7) |
| C(5)–C(6)–C(7) | 120.5(6) | C(15)–C(16)–C(17) | 119.6(7) |
| N(1)–C(7)–C(6) | 115.7(6) | C(12)–C(17)–C(16) | 120.2(7) |
| N(1)–C(7)–C(8) | 120.8(6) | O(1)–C(18)–C(19) | 109.2(8) |
| C(6)–C(7)–C(8) | 123.5(6) | | |

from a Patterson synthesis. In each of the three crystal structures the remaining non-hydrogen atoms were located by Fourier-difference syntheses. With the exception of the ring hydrogens of (2), which were located from a difference synthesis, H atoms were placed in calculated positions (C–H, 1.08 Å) and allowed to 'ride' on their respective carbon atoms in the subsequent least-squares refinements. Bond lengths and angles for (1), (2), and (3) are given in Tables 4—6 respectively and atomic co-ordinates in Tables 7—9 respectively.

Computations were carried out on the Birmingham University Honeywell computer with SHELX.[12] Stereoscopic views were drawn using PLUTO[13] at the University of Manchester regional computer centre.

tetrahydroborate reduction of 2-(quinolin-2-yl)phenyltellurium(IV) tribromide. This procedure gave the corresponding tritelluride in good yield. Two possible mechanisms, (1) and (2), may be considered for the formation of tritellurides [R = 2-(2-pyridyl)phenyl or 2-(quinolin-2-yl)phenyl].

$$2RTeBr_3 \xrightarrow{BH_4^-} 2RTeBr + Te^{2-} \longrightarrow R_2Te_3 + 2Br^- \quad (1)$$

$$\uparrow BH_4^-$$

$$Te$$

$$RTeBr_3 \xrightarrow{BH_4^-} RTe^- + Te \longrightarrow RTeTe^- \xrightarrow{O_2} R_2Te_3 \quad (2)$$

Mechanism (2) has been favoured by others,[22] but (1) may be applicable here given our practice of excluding air from our preparations. If this is so, we speculate that it may be possible to synthesise higher polytellurides, *e.g.* by reaction of a co-ordination stabilised organyltellurium halide with the ditelluride anion.

Reactions of 2-(2-pyridyl)phenyltellurium(IV) tribromide and of *p*-ethoxyphenyltellurium(IV) trichloride with 2-(2-pyridyl)-phenylmercury(II) chloride were carried out. It was anticipated that diorganyltellurium(IV) dihalides would be formed but the products contained both tellurium and mercury; further they were 1:1 electrolytes (see Table 1). The conductivities, together with the stoicheiometries, strongly suggest ionic structures $[R_2TeBr][HgClBr_2]$ and $[RR'TeCl][HgCl_3]$ respectively [R = 2-(2-pyridyl)phenyl, R′ = *p*-$EtOC_6H_4$]. It is interesting that the tellurium atom should bond to one pyridyl nitrogen atom rather than to the second halogen. For $[RR'TeCl][HgCl_3]$, a strong i.r. band at 287 $cm^{-1}$ is reasonably assigned as ν(TeCl).[26] Both compounds may be cleanly reduced to tellurides (Scheme). Bis[2-(2-pyridyl)phenyl] telluride may be converted to a diperchlorate salt.

*Spectroscopic Studies.*—I.r. and $^1H$ n.m.r. data were useful in confirming the metallation of 2-phenylpyridine and of 2-phenylquinoline; however, the $^{13}C$ n.m.r. data are generally more directly indicative of the position of metallation and the data in Table 2 are therefore worthy of brief comment. Mercuration of the bases causes a downfield shift of *ca.* 22 p.p.m. for one phenyl *ortho* carbon atom; similar observations are made for other mercurated species;[15,16,27] in this instance the rather limited solubility prevented the observation of $^{199}Hg$–$^{13}C$ coupling constants. The tellurium compounds are even less soluble, however spectra were obtained which confirmed the telluration of the bases, although the downfield shift of the metallated carbon is not quite so great. It is interesting that the resonances of the carbons *ortho* and *para* to nitrogen in the pyridine ring are more sensitive to the presence of tellurium than of mercury. This may reflect a stronger N–Te than N–Hg interaction. The spectrum of bis[2-(2-pyridyl)phenyl] tritelluride is of interest. The resonance of the tellurated carbon atom has shifted some 24 p.p.m. to higher field than in the corresponding mercurated derivative and than in the organyltellurium(IV) tribromide, (1). There is no value for the chemical shift of a similar carbon in the literature to provide a basis for comparison, however Zingaro and co-workers[20] have reported values of 123—125 p.p.m. for *ipso* carbons of some diorganyltellurium selenides. Again, a high-field (*ca.* 3 p.p.m.) shift for C(1) (adjacent to nitrogen, see Table 2) is seen which we take to be indicative of the relatively short Te ··· N contact of 2.55 Å in $R_2Te_3$.

The spectra of other compounds in Table 2 have been assigned following our previous work and the literature.[28,29]

*Structural Investigations.*—The structure of $RTeBr_3$ (1) [R = 2-(2-pyridyl)phenyl] is shown in Figure 1. The co-ordination about tellurium can be considered as essentially pseudo-octahedral, with a lone pair of electrons occupying the forth equatorial site. The whole molecule, apart from the apical bromine atoms, Br(1) and Br(2), is planar to within ±0.15 Å. Br(1) and Br(2) lie 2.739(5) and 2.575(3) Å on either side of the Te–N(1)–C(1)–Br(3) plane. The angle of 6.2(3)° between the mean planes of the pyridyl and phenyl rings illustrates the slight deviation from planarity of the organic ligand.

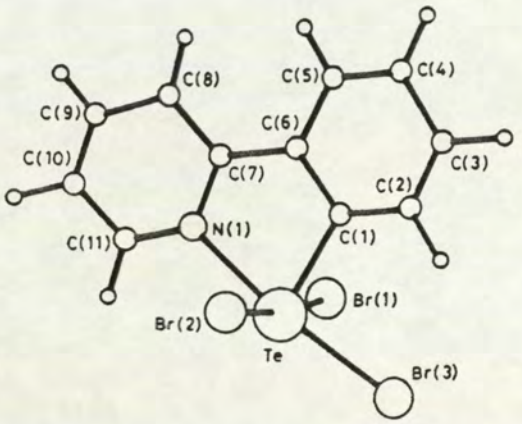



**Figure 1.** View of complex (1) showing the atom numbering

The distortion of the co-ordination geometry from ideal octahedral angles (90 and 180°), particularly N(1)–Te–Br(3) [170.4(4)°] can be explained in terms of a lone pair of electrons occupying an equatorial position between N(1) and Br(3). The small value of the angle N(1)–Te–C(1) [78.2(7)°] is due to the constraint of the five-membered chelate ring. The axial atoms are displaced from the lone pair electrons reducing the Br(1)–Te–Br(2) angle to 172.4(1)°. These values are analogous to those found in the crystal structure of (2-phenylazophenyl-*C*,*N*′)tellurium(IV) trichloride.[15]

The distance Te–C(1) [2.110(19) Å] is in excellent agreement with the sum of the Pauling[30] single bond covalent radii of Te (1.37 Å) and $sp^2$ hybridised carbon (0.74 Å) and with values in the crystal structures of the analogous compounds (pap)-$TeCl_3$ (pap = 2-phenylazophenyl),[15] (bip)$TeBr_3$ (bip = 2-biphenylyl),[31] and $C_4H_9STeBr_2$[32] in which Te–C is in the range 2.11—2.16 Å. The Te–N distance of 2.244(14) Å is longer than the sum of the covalent radii (2.07 Å),[30] but shorter than that reported for (2-phenylazophenyl-*C*,*N*′)tellurium(IV) trichloride,[15] 2.417 Å, and for (dapy)$TeCl_3$ (dapy = 2,6-diacetylpyridine), 2.402 Å,[33] indicating a stronger interaction in this case. The interaction between Te and the pyridyl nitrogen atom holds the organic ligand in an essentially planar geometry. The Te–Br distances [2.589(3) Å equatorial, 2.673(3) and 2.658(3) Å axial] are in good agreement with the sum of the covalent radii (equatorial Te–Br, 2.51 Å and axial Te–Br, 2.67 Å[30,34]) and fall within the range commonly found for Te–Br covalent bonds.[31,32,35] There is a weak secondary[36] intermolecular interaction between centrosymmetrically related molecules Te ··· Br of 3.596(3) Å, which is a feature noted in the structures of (bip)$TeBr_3$ (Te ··· Br, 3.71 Å)[31] and $C_4H_9STeBr_2$ (Te ··· Br, 3.59 Å).[32]

The structure of RTe(dmdtc) (2) [R = 2-(2-pyridyl)phenyl] is shown in Figure 2. The co-ordination about tellurium can be considered as essentially pseudo-trigonal bipyramidal, with C(1) and the lone pairs making up the equatorial co-ordination, and S(1)

**Table 7.** Fractional atomic co-ordinates ( × $10^4$) for compound (1) with e.s.d.s in parentheses

| Atom | *x* | *y* | *z* |
|---|---|---|---|
| Te | −1 148(2) | 4 702(1) | 6 901(1) |
| Br(1) | 373(3) | 3 242(3) | 8 772(2) |
| Br(2) | −2 577(4) | 6 522(3) | 5 122(2) |
| Br(3) | −3 547(4) | 2 395(3) | 7 016(3) |
| N(1) | 504(22) | 6 926(16) | 6 923(14) |
| C(1) | −3 102(29) | 5 931(23) | 7 944(17) |
| C(2) | −4 963(30) | 5 475(22) | 8 358(19) |
| C(3) | −5 976(34) | 6 289(29) | 9 061(19) |
| C(4) | −5 318(34) | 7 810(27) | 9 306(19) |
| C(5) | −3 481(28) | 8 252(22) | 8 884(16) |
| C(6) | −2 333(29) | 7 402(20) | 8 135(17) |
| C(7) | −467(27) | 7 924(21) | 7 633(16) |
| C(8) | 423(33) | 9 423(21) | 7 677(23) |
| C(9) | 2 327(35) | 9 709(22) | 7 082(25) |
| C(10) | 3 045(40) | 8 803(25) | 6 354(24) |
| C(11) | 2 176(31) | 7 331(28) | 6 366(18) |

**Table 8.** Fractional atomic co-ordinates for compound (2) ( × $10^5$ for Te, × $10^4$ for others) with e.s.d.s in parentheses

| Atom | *x* | *y* | *z* |
|---|---|---|---|
| Te | 105 314(3) | 13 542(3) | 33 091(3) |
| S(1) | 9 013(2) | 1 518(2) | 1 346(1) |
| S(2) | 5 995(2) | 3 887(2) | 2 727(1) |
| N(1) | 12 174(5) | 1 562(5) | 4 869(4) |
| N(2) | 5 861(5) | 2 549(6) | 713(4) |
| C(1) | 10 496(5) | 3 713(5) | 2 887(4) |
| C(2) | 9 645(6) | 4 791(6) | 1 842(5) |
| C(3) | 9 632(7) | 6 328(7) | 1 632(6) |
| C(4) | 10 470(7) | 6 795(6) | 2 417(6) |
| C(5) | 11 331(7) | 5 731(6) | 3 463(5) |
| C(6) | 11 368(6) | 4 187(5) | 3 690(4) |
| C(7) | 12 295(6) | 2 992(6) | 4 743(4) |
| C(8) | 13 305(8) | 3 254(7) | 5 579(5) |
| C(9) | 14 156(9) | 2 030(10) | 6 489(6) |
| C(10) | 14 017(7) | 540(8) | 6 609(5) |
| C(11) | 13 012(7) | 361(7) | 5 768(5) |
| C(12) | 6 811(6) | 2 680(5) | 1 556(4) |
| C(13) | 3 998(8) | 3 513(9) | 714(6) |
| C(14) | 6 548(9) | 1 467(10) | −296(6) |

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom co-ordinates and thermal parameters.

## Discussion

2-Phenylpyridine and 2-phenylquinoline have both been directly *ortho*-palladated when treated with $Na_2[PdCl_4]$ in ethanol.[14] Although the direct telluration of azobenzene with tellurium tetrachloride can proceed under forcing conditions,[15] generally a *trans* metallation route must be used for the synthesis of *ortho*-tellurated bases.[16] The mercuration of 2-phenylpyridine and 2-phenylquinoline was achieved *via* mercury(II) acetate, generally considered a stronger electrophile than mercury(II) chloride.[17] A method similar to that for the mercuration of azobenzene[18] was adopted and gave moderate, but adequate yields, of the mercurated products. The telluration was achieved by *trans* metallation with $TeBr_4$ (which is less readily hydrolysed than $TeCl_4$ and gives fewer problems with protonated by-products) or with (*p*-ethoxyphenyl)tellurium(IV) trichloride (Table 1 and Scheme).

Reduction of the tribromides with the stoichiometric quantity of hydrazine hydrate affords the organyltellurium

**Table 9.** Fractional atomic co-ordinates for compound (3) ( × $10^5$ for Te, × $10^4$ for others) with e.s.d.s in parentheses

| Atom | *x* | *y* | *z* |
|---|---|---|---|
| Te | 12 013(3) | −2 182(3) | −13 169(5) |
| O(1) | 3 726(4) | 1 292(3) | 4 833(6) |
| N(1) | 445(4) | −1 079(4) | −4 005(7) |
| C(1) | 2 325(4) | −1 013(4) | −2 275(8) |
| C(2) | 3 301(5) | −944(4) | −1 675(8) |
| C(3) | 4 041(5) | −1 425(5) | −2 353(10) |
| C(4) | 3 803(6) | −1 975(5) | −3 648(10) |
| C(5) | 2 858(5) | −2 068(5) | −4 222(9) |
| C(6) | 2 075(5) | −1 595(4) | −3 562(8) |
| C(7) | 1 049(5) | −1 707(4) | −4 209(7) |
| C(8) | 708(6) | −2 422(5) | −4 983(10) |
| C(9) | −256(6) | −2 471(6) | −5 548(11) |
| C(10) | −880(6) | −1 825(6) | −5 362(10) |
| C(11) | −496(6) | −1 138(5) | −4 562(10) |
| C(12) | 2 110(5) | 246(4) | 773(8) |
| C(13) | 2 428(5) | 1 037(5) | 739(9) |
| C(14) | 2 966(5) | 1 380(4) | 2 097(9) |
| C(15) | 3 208(5) | 911(5) | 3 525(9) |
| C(16) | 2 912(6) | 114(5) | 3 606(10) |
| C(17) | 2 357(6) | −224(5) | 2 220(9) |
| C(18) | 3 971(6) | 835(6) | 6 357(10) |
| C(19) | 4 596(8) | 1 331(6) | 7 514(13) |

bromides which are stabilised by co-ordination of the heterocyclic nitrogen atom; these materials undergo metathesis with sodium dimethyldithiocarbamate to give the organyltellurium(II) dithiocarbamates (see later).

The reduction of both tribromides with an excess of hydrazine hydrate gives the corresponding ditellurides. In principle, the reduction of a ditelluride with sodium tetrahydroborate under mildly alkaline conditions should give the sodium organyltellurate, a useful nucleophile. Attempts were made to eliminate intermediate stages of the synthesis by reacting the tribromides directly with sodium tetrahydroborate. For the 2-phenylquinoline series only the ditelluride was isolated; however in the 2-phenylpyridine series an initial precipitate of tellurium redissolved during the course of the reaction. Work-up gave a novel diorganyl tritelluride the nature of which was confirmed by *X*-ray crystallography.* Although triselenides have been known for some time,[19] and Zingaro and co-workers[20,21] have reported a range of diorganyltellurium selenides, the tritellurides reported here, together with $(Me_3Si)_3CTeTeTeC(SiMe_3)_2$[22] constitute the first well characterised examples of diorganyl tritellurides.

The stabilisation of bis[tris(trimethylsilyl)methyl] tritelluride[22] has been attributed to the bulk of the organic group. The compounds in this paper owe their stability to co-ordination of the heterocyclic nitrogen atom to the terminal tellurium atoms (Te–N, 2.554 Å; *cf.* van der Waals distance of 3.61 Å). The Te–Te bond length (2.776 Å) is longer than that for some ditellurides, *e.g.* $Ph_2Te_2$ (2.712 Å),[23] $(p\text{-}CH_3C_6H_4)_2Te_2$ (2.697 Å),[24] and $(p\text{-}MeOC_6H_4)_2Te_2$ (2.72 Å)[25] and it is also longer by 0.066 Å than the Te–Te distance in bis[tris(trimethylsilyl)-methyl] tritelluride.[22] It is likely that a wide range of diorganyl polytellurides may be stabilised by co-ordination of the terminal tellurium atoms.

Reduction of 2-(2-pyridyl)phenyltellurium(IV) tribromide, (1), with an excess of hydrazine hydrate gave bis[2-(2-pyridyl)phenyl] ditelluride (Scheme); also treatment of the tritelluride with copper powder gave the same ditelluride. The observation that an initial precipitate of tellurium seemed responsible for the formation of the tritelluride on $NaBH_4$ reduction of (1) led us deliberately to add tellurium powder in a repeat of the

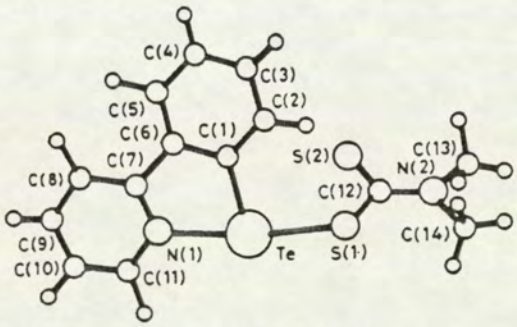



Figure 2. View of complex (2) showing the atom numbering

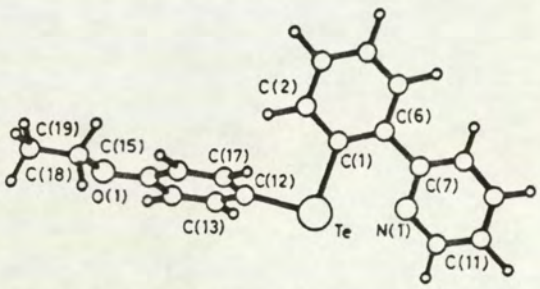



Figure 3. View of complex (3) showing the atom numbering

and N(1) axial. The position *trans* to the bonded carbon atom is unoccupied and the compound is almost 'T'-shaped.[34] The 2-(2-pyridyl)phenyl ligand is planar to within ±0.06 Å, with the Te atom displaced 0.03 Å from the plane, and the S(1) atom also displaced 0.30 Å. The dimethyldithiocarbamato group is planar to within ±0.04 Å and is orientated at an angle of 72.7(2)° to the Te–R plane. The distortion of the co-ordination geometry from ideal pseudo-trigonal bipyramidal values, particularly the N(1)–Te–C(1) angle of 74.4(2)°, is again due to constraints arising from the five-membered chelate ring.

The distance Te–C(1) [2.111(5) Å] is in good agreement with the sum of the Pauling covalent single bond radii[30] and with typical values in $Te^{II}$ complexes, *e.g.* $PhTe(tu)_2Cl$,[37] PhTe(tmsu)Cl,[37] and (pap)Te(dmdtc)[38] (where tu = thiourea and tmsu = *N*,*N*′-trimethyleneselenourea), which lie in the range 2.097—2.102 Å. The Te–N distance, 2.354(4) Å, is longer than the sum of the covalent radii (2.24 Å for axial Te–N)[30,34] and is also longer than in (pap)Te(SCN), 2.243 Å,[39] and in (pap)TeCl, 2.23 Å,[40] but is comparable with that reported for (pap)Te(dmdtc), 2.340 Å.[35] The Te–N interaction holds the organic ligand is an almost planar geometry, with only an angle at 4.4(2)° between the mean planes of the pyridyl and phenyl rings. The Te–S(1) bond, 2.518(1) Å, is in good agreement with the sum of the S and Te axial covalent radii,[30,34] 2.58 Å, and similar to values found in other tellurium(II) complexes with sulphur ligands,[37] *e.g.* $Te(S_2COR)_2$ (R = Me or Et), $Te(S_2CNCH_2CH_2OCH_2CH_2)_2$, and $Te(S_2CNEt_2)_2$, which fall in the range 2.49—2.52 Å. The Te–S(2) distance of 3.667(1) Å, although within the sum of the van der Waals radii (4.05[30] or 3.86 Å[41]), indicates only an extremely weak interaction. By contrast, dimethyldithiocarbamato(2-phenylazophenyl-*C*,*N*′)-tellurium(II) has both sulphur atoms essentially coplanar with the tellurium atoms and the bonded C and N atoms of the organic ligand and the Te–S(2) distance of 3.225(3) Å clearly shows a significantly stronger interaction in the case of the tellurated azobenzene derivative.

The arrangement of the molecules in the unit cell reveals that almost planar organic ligands related by centres of symmetry overlie one another, with an interplanar distance of 3.50(1) Å. No unusually short intermolecular contacts occur.

The structure of $RTe(C_6H_4OEt\text{-}p)$ [R = 2-(2-pyridyl)-phenyl] is shown in Figure 3. The tellurium atom is bonded to two carbon atoms with Te–C(1) 2.138(6) and Te–C(12) 2.144(6) Å, in good agreement with the sum of the Pauling[30] single bond covalent radii and with values in the crystal structures of analogous compounds, *e.g.* $R_2Te_3$ [R = 2-(2-pyridyl)phenyl],[1] 1,6-bis(2-butyltellurophenyl)-2,5-diazahexa-1,5-diene[2] and in complexes (1) and (2) above, which lie in the range 2.10—2.13 Å. The Te–N distance of 2.695(4) Å is much greater than the sum of the covalent radii[30] and much greater than the comparable distance in (1) and (2) above. The occurrence of a Te–C covalent bond *trans* to the Te ··· N vector thus significantly weakens the Te–N interaction. A similar Te ··· N distance of 2.773 Å occurs in a previously reported bis-telluride.[2] The weakening of the Te–N interation allows the rings in the 2-(2-pyridyl)phenyl moiety to twist by 23.2(2)° relative to each other, unlike in (1) and (2) where the stronger Te–N bonding holds the corresponding organic ligand in a near-planar geometry. Within the *p*-ethoxyphenyl ligand, the ethoxy group is orientated at 4.3(1)° to the phenyl ring. No unusually short intermolecular contacts occur.

Vikane[42] has noted, with reference to organyltellurium bromide complexes, that only long weak interactions are found *trans* to a Te–C bond. A similar effect has been noted in organyltellurium(IV) bromides and iodides;[31,32,35] strong Te–halogen interactions are never *trans* to a Te–C bond. It is also true of complexes (1), (2), and (3) that no strong bonds are seen *trans* to a Te–C covalent bond.

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# The crystal and molecular structure of dimethyldithiocarbamato-2-(2-quinolinyl)phenyltellurium(II)

**Antony A. West, William R. McWhinnie,**

*Department of Chemical Engineering and Applied Chemistry, University of Aston in Birmingham, Aston Triangle, Birmingham B4 7ET (Great Britain)*

**and Thomas A. Hamor**

*Department of Chemistry, University of Birmingham, Birmingham B15 2TT (Great Britain)*

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## Abstract

The crystal structure of dimethyldithiocarbamato-2(2-quinolinyl)phenyltellurium (II) has been determined from three dimensional X-ray counter data. The crystal system is triclinic, space group $P\bar{1}$ with $a$ 7.360(5), $b$ 15.022(10), $c$ 17.730(10) Å, $\alpha$ 114.26(2)°, $\beta$ 94.12(6)°, $\gamma$ 95.63(3)°, $Z = 4$, $R$ 3.36% for 4969 observed reflections; For two independent molecules, the coordination about Te can be considered to be trigonal bipyramidal, with a carbon atom and the two lone pairs of Te comprising the equatorial coordination, and sulphur together with the quinolinyl nitrogen atom in axial positions. (Te · · · C 2.124(5), 2.135(4); Te · · · N 2.365(4), 2.385(4); Te · · · S(1) 2.569(1), 2.543(1) Å). S(2) makes a weak secondary interaction with Te (3.222(1), 3.230(1) Å) in the C,N,S(1) plane. There are no significant intermolecular contacts.

## Introduction

The preparation and chemical properties of the title compound have previously been reported by Al-Salim et al. [1]. Following earlier structural studies of analogous complexes in which short Te–N interactions were found [1,2], we now examine the effect of the tellurium oxidation state and of the type of ligand *trans* to nitrogen on the Te···N interaction.

## X-ray structure analysis

After a preliminary study by photographic methods, cell parameters and reflection intensities were measured with graphite monochromated Mo-$K_\alpha$ radiation from

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a yellow/orange crystal, 0.60 × 0.30 × 0.25 mm, using an Enraf–Nonius CAD-4 diffractometer, operating in the $\omega-2\theta$ scan mode. Three standard reflections were monitored at regular intervals to check the stability of the system. 6400 reflections were scanned in the range $2 < \theta < 25°$. Lorentz and polarization factors were applied to the data, and 4969 unique reflections with $I > 2.5\sigma(I)$ were used in the structural analysis. The crystals were triclinic with no systematic absences, the centrosymmetric space group $P\bar{1}$ being established by the successful analysis.

*Crystal data*

$C_{18}H_{16}N_2S_2Te$, $M_r = 452.1$, space group $P\bar{1}$, $a$ 7.360(5), $b$ 15.022(10), $c$ 17.730(10) Å, $\alpha$ 114.26(2)°, $\beta$ 94.12(6)°, $\gamma$ 95.63(3)°, $V$ 1764.9 Å$^3$, $Z = 4$, $D_c$ 1.701 g cm$^{-3}$, $F(000) = 888$, $\lambda$(Mo-$K_\alpha$) 0.71069, $\mu$ 1.97 mm$^{-1}$.

The atomic coordinates for tellurium and for most of the lighter atoms were found by direct methods using SHELX86 [3]. The remaining non-hydrogen atoms were located by a Fourier difference synthesis. As only some of the hydrogens could be located from difference maps, all the hydrogen atoms were placed in calculated positions (C–H 1.08 Å) and allowed to "ride" on their respective carbon atoms in the subsequent least-squares refinements. The weighting scheme used was $w = 1/[\sigma^2(F) + 0.0006F^2]$ and the refinements converged to $R = 0.0336$ and $R_w = 0.0485$, when all shifts were $< 0.1\sigma$.

Non-hydrogen atomic coordinates are listed in Table 1 and bond lengths and angles in Table 2. Computations were carried out on the Birmingham University

Table 1

Fractional atomic coordinates ($\times 10^5$ for tellurium and $\times 10^4$ for all other non-hydrogen atoms) with e.s.d.'s in parentheses

| | $x$ | $y$ | $z$ | | $x$ | $y$ | $z$ |
|---|---|---|---|---|---|---|---|
| Te(1A) | 29856(3) | 73451(2) | 222(2) | Te(1B) | 28125(3) | 72503(2) | 50311(2) |
| S(1A) | 3022(2) | 9211(1) | 507(1) | S(1B) | 1843(2) | 8876(1) | 5985(1) |
| S(2A) | 2202(2) | 8190(1) | −1354(1) | S(2B) | −882(2) | 7902(1) | 4443(1) |
| N(1A) | 2858(4) | 5744(2) | −24(2) | N(1B) | 4565(5) | 5930(3) | 4422(2) |
| N(2A) | 2200(5) | 10102(2) | −448(2) | N(2B) | −1156(5) | 9572(2) | 5738(2) |
| C(1A) | 3740(5) | 7560(3) | 1271(3) | C(1B) | 5187(6) | 7549(3) | 5913(3) |
| C(2A) | 4352(6) | 8476(3) | 1926(3) | C(2B) | 5444(7) | 8327(4) | 6694(3) |
| C(3A) | 4865(7) | 8556(4) | 2721(3) | C(3B) | 7043(8) | 8525(4) | 7220(4) |
| C(4A) | 4735(7) | 7730(5) | 2884(3) | C(4B) | 8395(9) | 7945(5) | 6965(4) |
| C(5A) | 4125(7) | 6813(4) | 2245(3) | C(5B) | 8182(7) | 7156(4) | 6200(4) |
| C(6A) | 3641(5) | 6713(3) | 1428(3) | C(6B) | 6532(6) | 6940(4) | 5663(3) |
| C(7A) | 3074(5) | 5747(3) | 729(3) | C(7B) | 6204(6) | 6086(3) | 4845(3) |
| C(8A) | 2732(7) | 4844(4) | 813(3) | C(8B) | 7529(8) | 5427(4) | 4501(4) |
| C(9A) | 2150(6) | 4003(4) | 145(3) | C(9B) | 7076(9) | 4660(5) | 3775(5) |
| C(10A) | 1897(5) | 3977(3) | −667(3) | C(10B) | 5288(8) | 4466(4) | 3294(4) |
| C(11A) | 2302(5) | 4891(3) | −722(3) | C(11B) | 4050(7) | 5142(3) | 3662(3) |
| C(12A) | 2151(7) | 4898(3) | −1517(3) | C(12B) | 2274(7) | 4985(3) | 3233(3) |
| C(13A) | 1547(8) | 4042(3) | −2222(3) | C(13B) | 1722(9) | 4191(3) | 2487(3) |
| C(14A) | 1078(7) | 3164(4) | −2162(4) | C(14B) | 2986(11) | 3525(4) | 2135(4) |
| C(15A) | 1273(7) | 3123(3) | −1407(4) | C(15B) | 4709(10) | 3665(4) | 2523(4) |
| C(16A) | 2452(5) | 9221(3) | −465(3) | C(16B) | −199(6) | 8824(3) | 5387(3) |
| C(17A) | 1825(7) | 10215(4) | −1225(3) | C(17B) | −2875(7) | 9632(4) | 5307(4) |
| C(18A) | 2392(6) | 11002(3) | 319(3) | C(18B) | −5756(7) | 10369(3) | 5577(3) |

Table 2

Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses

| | | | |
|---|---|---|---|
| Te(1A)–S(1A) | 2.569(1) | Te(1B)–S(1B) | 2.543(1) |
| Te(1A)...S(2A) | 3.222(1) | Te(1B)...S(2B) | 3.230(1) |
| Te(1A)–N(1A) | 2.365(4) | Te(1B)–N(1B) | 2.385(4) |
| Te(1A)–C(1A) | 2.124(5) | Te(1B)–C(1B) | 2.135(4) |
| S(1A)–C(16A) | 1.751(5) | S(1B)–C(16B) | 1.752(5) |
| S(2A)–C(16A) | 1.677(40 | S(2B)–C(16B) | 1.676(4) |
| N(1A)–C(7A) | 1.331(7) | N(1B)–C(7B) | 1.320(6) |
| N(1A)–C(11A) | 1.365(4) | N(1B)–C(11B) | 1.373(5) |
| N(2A)–C(16A) | 1.343(6) | N(2B)–C(16B) | 1.338(5) |
| N(2A)–C(17A) | 1.465(8) | N(2B)–C(17B) | 1.461(7) |
| N(2A)–C(18A) | 1.456(5) | N(2B)–C(18B) | 1.473(5) |
| C(1A)–C(2A) | 1.390(5) | C(1B)–C(2B) | 1.381(6) |
| C(1A)–C(6A) | 1.407(8) | C(1B)–C(6B) | 1.386(6) |
| C(2A)–C(3A) | 1.385(8) | C(2B)–C(3B) | 1.375(8) |
| C(3A)–C(4A) | 1.381(10) | C(3B)–C(4B) | 1.366(9) |
| C(4A)–C(5A) | 1.381(7) | C(4B)–C(5B) | 1.371(7) |
| C(5A)–C(6A) | 1.409(8) | C(5B)–C(6B) | 1.411(7) |
| C(6A)–C(7A) | 1.465(5) | C(6B)–C(7B) | 1.472(6) |
| C(7A)–C(8A) | 1.424(8) | C(7B)–C(8B) | 1.443(8) |
| C(8A)–C(9A) | 1.331(6) | C(8B)–C(9B) | 1.320(8) |
| C(9A)–C(10A) | 1.421(9) | C(9B)–C(10B) | 1.449(9) |
| C(10A)–C(11A) | 1.420(7) | C(10B)–C(11B) | 1.418(7) |
| C(10A)–C(15A) | 1.412(6) | C(10B)–C(15B) | 1.402(7) |
| C(11A)–C(12A) | 1.410(8) | C(11B)–C(12B) | 1.411(7) |
| C(12A)–C(13A) | 1.379(6) | C(12B)–C(13B) | 1.367(6) |
| C(13A)–C(14A) | 1.378(8) | C(13B)–C(14B) | 1.417(9) |
| C(14A)–C(15A) | 1.363(10) | C(14B)–C(15B) | 1.352(10) |
| | | | |
| S(1A)–Te(1A)–N(1A) | 163.7(1) | S(1B)–Te(1B)–N(1B) | 162.8(1) |
| S(1A)–Te(1A)–C(1A) | 90.3(1) | S(1B)–Te(1B)–C(1B) | 89.0(1) |
| N(1A)–Te(1A)–C(1A) | 74.5(1) | N(1B)–Te(1B)–C(1B) | 74.0(1) |
| Te(1A)–S(1A)–C(16A) | 98.6(1) | Te(1B)–S(1B)–C(16B) | 99.1(1) |
| Te(1A)–N(1A)–C(7A) | 113.1(2) | Te(1B)–N(1B)–C(7B) | 112.7(3) |
| Te(1A)–N(1A)–C(11A) | 124.7(3) | Te(1B)–N(1B)–C(11B) | 124.7(3) |
| C(7A)–N(1A)–C(11A) | 121.3(4) | C(7B)–N(1B)–C(11B) | 122.1(4) |
| C(16A)–N(2A)–C(17A) | 120.6(3) | C(16B)–N(2B)–C(17B) | 121.2(3) |
| C(16A)–N(2A)–C(18A) | 123.2(4) | C(16B)–N(2B)–C(18B) | 122.8(4) |
| C(17A)–N(2A)–C(18A) | 116.0(4) | C(17B)–N(2B)–C(18B) | 116.0(4) |
| Te(1A)–C(1A)–C(2A) | 124.1(4) | Te(1B)–C(1B)–C(2B) | 123.1(4) |
| Te(1A)–C(1A)–C(6A) | 117.0(3) | Te(1B)–C(1B)–C(6B) | 117.1(3) |
| C(2A)–C(1A)–C(6A) | 118.9(5) | C(2B)–C(1B)–C(6B) | 119.8(4) |
| C(1A)–C(2A)–C(3A) | 120.8(5) | C(1B)–C(2B)–C(3B) | 120.9(5) |
| C(2A)–C(3A)–C(4A) | 120.6(4) | C(2B)–C(3B)–C(4B) | 119.4(5) |
| C(3A)–C(4A)–C(5A) | 119.7(6) | C(3B)–C(4B)–C(5B) | 121.5(5) |
| C(4A)–C(5A)–C(6A) | 120.5(6) | C(4B)–C(5B)–C(6B) | 119.3(5) |
| C(1A)–C(6A)–C(5A) | 119.4(4) | C(1B)–C(6B)–C(5B) | 119.0(4) |
| C(1A)–C(6A)–C(7A) | 118.7(5) | C(1B)–C(6B)–C(7B) | 119.3(4) |
| C(5A)–C(6A)–C(7A) | 121.9(5) | C(5B)–C(6B)–C(7B) | 121.7(4) |
| N(1A)–C(7A)–C(6A) | 115.9(5) | N(1B)–C(7B)–C(6B) | 115.9(4) |
| N(1A)–C(7A)–C(8A) | 119.7(4) | N(1B)–C(7B)–C(8B) | 119.8(4) |
| C(6A)–C(7A)–C(8A) | 124.4(5) | C(6B)–C(7B)–C(8B) | 124.3(4) |
| C(7A)–C(8A)–C(9A) | 120.2(6) | C(7B)–C(8B)–C(9B) | 119.6(5) |
| C(8A)–C(9A)–C(10A) | 121.3(6) | C(8B)–C(9B)–C(10B) | 122.0(6) |
| C(9A)–C(10A)–C(11A) | 116.3(4) | C(9B)–C(10B)–C(11B) | 115.8(4) |
| C(9A)–C(10A)–C(15A) | 125.1(5) | C(9B)–C(10B)–C(15B) | 125.1(6) |
| C(11A)–C(10A)–C(15A) | 118.5(5) | C(11B)–C(10B)–C(15B) | 119.1(5) |

(continued)

Table 2 (continued)

| | | | |
|---|---|---|---|
| N(1A)–C(11A)–C(10A) | 121.0(5) | N(1B)–C(11B)–C(10B) | 120.7(4) |
| N(1A)–C(11A)–C(12A) | 120.4(4) | N(1B)–C(11B)–C(12B) | 120.8(4) |
| C(10A)–C(11A)–C(12A) | 118.6(4) | C(10B)–C(11B)–C(12B) | 118.5(4) |
| C(11A)–C(12A)–C(13A) | 120.6(5) | C(11B)–C(12B)–C(13B) | 121.6(5) |
| C(12A)–C(13A)–C(14A) | 120.6(6) | C(12B)–C(13B)–C(14B) | 118.7(6) |
| C(13A)–C(14A)–C(15A) | 120.4(4) | C(13B)–C(14B)–C(15B) | 121.2(5) |
| C(10A)–C(15A)–C(14A) | 121.3(5) | C(10B)–C(15B)–C(14B) | 120.8(6) |
| S(1A)–C(16A)–S(2A) | 121.8(3) | S(1B)–C(16B)–S(2B) | 121.9(3) |
| S(1A)–C(16A)–N(2A) | 115.6(3) | S(1B)–C(16B)–N(2B) | 115.2(3) |
| S(2A)–C(16A)–N(2A) | 122.6(4) | S(2B)–C(16B)–N(2B) | 122.9(3) |

Honeywell computer and at the University of Manchester Regional Computer Centre with SHELX76 [4] and SHELX86 [3]; Stereoscopic views were drawn using PLUTO [5].

## Discussion

The molecular structure is shown in Fig. 1. The structure contains two crystallographically independent molecules within the asymmetric unit. The coordination about tellurium can be considered as essentially $\psi$-trigonal bipyramidal, with C(1) and the two lone pairs of Te making up the equatorial coordination, and with S(1) and N(1) axial. The distortion of coordination geometry from ideal $\psi$-trigonal bipyramidal, particularly N(1)–Te–C(1) (74.5(1)° molecule A, 74.0(1)° molecule B) is due to the constraint imposed by the five-membered chelate ring. S(2) makes a weak secondary interaction in the C(1),N(1),S(1) plane, which bisects the angle between the lone pairs. Thus the position *trans* to the bonded phenyl carbon is unoccupied, and the compound can be regarded as almost "T-shaped" [6].

The 2-(2-quinolinyl)phenyl ligand is planar to within ±0.15 Å in molecule A and ±0.10 Å in molecule B, with a twist of 8.3(3)°, 5.3(2)° between the mean planes of

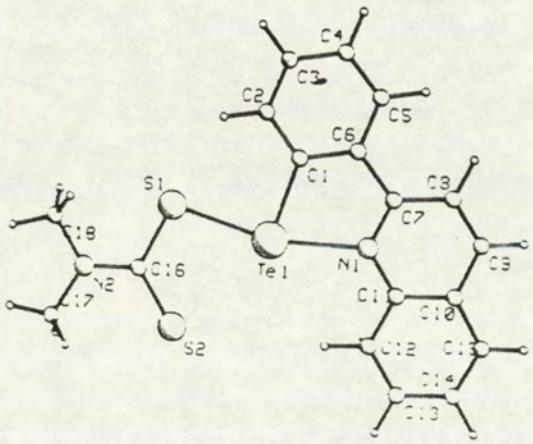


Fig. 1. View of complex A showing the atom numbering. The other molecule in the asymmetric unit (complex B) has virtually identical conformation.

the quinolinyl and phenyl rings. The tellurium atom is displaced 0.33 Å (for A) and 0.38 Å (for B) from this plane; S(1) is displaced by 0.94 Å and 0.78 Å and S(2) by 0.80 Å and 1.07 Å for molecules A and B, respectively. The dimethyldithiocarbamate group is planar and is oriented at angles of 12.0(4)°, 3.9(2)° to the chelate ring plane in the two molecules.

The distance Te–C(1) (2.124(5), 2.135(4) Å) is in good agreement with the sum of the Pauling single bond covalent radii (2.11 Å) [7] and with values reported for analogous $Te^{II}$ complexes, $Te(C_5H_4NPh)(dmdtc)$ [1], $TePh(tu)_2Cl$ [8] and $Te(C_6H_4N_2Ph)(dmdtc)$ [9] (where dmdtc = dimethyldithiocarbamate and tu = thiourea), which lie in the range 2.101–2.111 Å. The Te–N distance (2.365(4), 2.385(4) Å) is larger than the sum of the covalent radii (2.24 Å for axial Te–N) [6,7] and longer than that reported for the same interaction in $Te(C_6H_4N_2Ph)(SCN)$ [2.243 Å] [10] and $Te(C_6H_4N_2Ph)Cl$ (2.230 Å) [11], but compares well with the values reported for $Te(C_6H_4N_2Ph)(dmdtc)$ (2.340 Å) [9] and $Te(C_5H_4NPh)(dmdtc)$ (2.354 Å) [1]. The Te–N interaction holds the organic ligand in the almost planar geometry, noted above.

The Te–S(1) bond length (2.569(1) Å, 2.543(1) Å) is in good agreement with the sum of the S and Te axial covalent radii (2.58 Å) [6,7] and with values found in other tellurium(II) complexes containing sulphur ligands, e.g. $Te(C_6H_4N_2Ph)(dmdtc)$ [9], $Te(C_5H_4NPh)(dmdtc)$ [1] and $Te(Et_2NCS_2)_2$ [12], which fall in the range 2.52–2.57 Å. Compared with (2-phenylazophenyl-*C*,*N*′)thiocyanatotellurium(II) (Te–C 2.073 Å, Te–N 2.243 Å and Te–S 2.672 Å) [10], the bonding to the ligand is somewhat weaker and that to the sulphur somewhat stronger.

The Te–S(2) distance (3.222(1) Å, 3.230(1) Å), although within the sum of the Van der Waals radii (4.05 Å) [7] or (3.86 Å) [13], seems to indicate only a weak secondary interaction between these atoms, which is a feature noted in $Te(C_6H_4N_2Ph)(dmdtc)$ (Te...S(2) 3.225 Å) [9].

The approach to the tellurium by a fourth atom, S(2) of the dimethyldithiocarbamate group, could be considered to be a manifestation of the tendency of $Te^{II}$ to achieve a square planar geometry, as has been reported for many $Te^{II}$ complexes. In this compound both the sulphur atoms are essentially coplanar with the tellurium atom and the coordinating C and N atoms of the organic ligand. This situation is akin to that found in dimethyldithiocarbamato(2-phenylazophenyl-*C*,*N*′)tellurium(II) [9], but in contrast to that in dimethyldithiocarbamato(2-[2-pyridyl]phenyl)tellurium(II) [1], where the dimethyldithiocarbamate group is oriented at an angle of 72.7(3)° to the tellurium-organic ligand plane and there is considered to be no significant interaction between Te and S(2) (Te...S(2) 3.667(1) Å) [1].

As pointed out earlier by Foss et al. [14,15], the *trans* effect of the phenyl group in three coordinate tellurium(II) complexes is pronounced. In our compound we have the typical "T-shape" found for three coordinate tellurium(II) complexes, with the phenyl group at the stem of the "T", and no strong interactions *trans* to the phenyl group. This is consistent with results we described previously [1,2] and with the work of Vikane [16].

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