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# STUDIES ON TELLURIUM-CARBON BONDED COMPOUNDS

## V*. THE CRYSTAL STRUCTURE OF 1,3-DIMETHYLACETYLACETONE TELLURIUM(II)

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## Summary

The structure of the title compound has been determined by the heavy-atom method from 1349 reflections, measured with a diffractometer, and refined by full-matrix least-squares methods to $R$ 0.044. Crystals are monoclinic, space group $B2_1/c$, $a = 23.53(3)$, $b = 4.85(1)$, $c = 16.10(2)$ Å, $\beta = 111.5(1)°$, $Z = 8$. In the molecules of the complex a tellurium(II) atom is bonded to one bivalent 1,3-dimethylacetylacetone ligand via the α-carbon atoms [Te–C 2.202(8), 2.157(8) Å; C–Te–C 88.4(3)°] forming a heterocyclic ring of chair conformation with both methyl substituents in equatorial positions. The molecules are arranged into one-dimensional zig-zag polymers, about the $2_1$ axes at $x = \frac{1}{4}$ or $\frac{3}{4}$ and $z = 0$ or $\frac{1}{2}$, by associations between the tellurium atoms. These associations have been observed in other compounds studied in previous parts of this series and the implications of these associations with regard to the colour of this and related tellurium compounds are investigated in the light of current theories. All hydrogen atoms have been located.

## Introduction

Crystal structures of many acetylacetone (acac) complexes have

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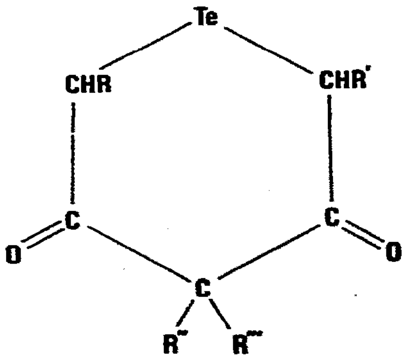
[*] For part IV see ref.18.

been investigated by X-ray analysis in recent years [1-13], and in the majority of these the ligand behaves as a univalent bidentate ligand bonding via the oxygen atoms [1-7]. Only in a few cases, in certain compounds with platinum(II), platinum(IV) and sulphur, has the mode of attachment of the ligand been shown to be anomalous [8-13]. In $(acac)_2S_2$, two (acac) ligands bond to either atom of a disulphur molecule via the γ-carbon atoms [8]. In the case of platinum [9-13], (acac) has been found to be either uni- or tri-functional. In the unifunctional case [9-11] the ligand bonds to platinum solely via the γ-carbon in a similar manner to that found for sulphur. When the ligand is trifunctional [12,13] it bonds via the carbonyl oxygens to one platinum and to another platinum via the γ-carbon.



(I) : $R = R' = H$ ; $R'' = R''' = CH_3$
(II) : $R = R' = CH_3$; $R'' = R''' = H$
(III) : $R = R' = R'' = R''' = H$
(IV) : $R = R' = R'' = CH_3$ ; $R''' = H$
(V) : $R = R'' = CH_3$ ; $R' = R''' = H$

We have previously reported N.M.R. data [14], and the crystal structures of 3,3-dimethylacetylacetone tellurium(II), (I) [15]; 1,1'-dimethylacetylacetone tellurium(II), (II) [16]; acetylacetone tellurium(II), (III) [17]; and 1,3,1'-trimethylacetylacetone tellurium(II), (IV) [18], wherein the β-diketone acts as a bivalent bidentate chelate bonding to the tellurium(II) atom via the α-carbon atoms. This is the first time that acetylacetone and its derivatives have been shown, by reported by Professor Sir Gilbert T. Morgan and co-workers [19-26] as part of their classical researches in coordination chemistry at the University of Birmingham (U.K.) in the early 1920's. The only other crystal structure of a tellurium(II) organometallic compound containing a bivalent bidentate chelate is that of phenoxatellurine [27].

In compounds (II-IV) [16-18] the molecules have been found to be associated with one another by tellurium-tellurium contacts (of ca. 4 Å) that form one-dimensional polymeric chains of tellurium atoms lying along the major growth directions of the crystals. McCullough [28-31] has previously noted the significance of associations such as I-Te...I in tellurium(IV) structures and how specific associations seem to give rise to particular colours. Donaldson and Silver [32-35] have recently connected colour and electrical properties, in compounds of p-block elements, with donation of electron density from non-bonding lone pair orbitals into 'bands' in the compound, and we have used similar arguments to explain the colours of Morgan's compounds [19-26] and also those studied by McCullough [28-31]. There is evidence, both crystallographic [36] and spectroscopic [37], that tellurium compounds containing no associations at all are colourless. As a continuation of our studies in this area, we here report the crystal structure of 1,3-dimethylacetylacetone tellurium(II),(V).

## Experimental

### Data Collection

Crystals suitable for X-ray work were obtained from the museum in the Chemistry Department of this University and are almost certainly originals from Morgan's time [19-26].

Approximate cell dimensions were determined by oscillation, Weissenberg and precession photographs, final cell dimensions being measured on a Stoe two-circle computer-controlled semi-automatic diffractometer with graphite monochromated Mo-$K_\alpha$ radiation and a scintillation counter. Intensity data moving-crystal method being used, with an ω-scan range of 2.0° on the lower levels, counting for 0.15 s at 0.01° intervals. This corresponds to a scan rate of 4°/min. For reflections on the second and higher layers ($\mu > 7°$) a variable range was scanned, with $\Delta\omega$ defined by $(A + B \sin \mu/\tan \theta')°$ where $2\theta'$ is the azimuth angle [38] and $A = 1.0$ and $B = 0.5$. Stationary-background counts were measured at the extremities of each scan for 10 s. Within the range $0.1 < \sin \theta/\lambda \leqslant 0.65$, 1349 independent reflections having $I > 2.5\ \sigma(I)$ were obtained and considered observed. Data were corrected for Lorentz and polarization factors but not for absorption, since the crystal was a needle of uniform cross-section elongated parallel to the $b$ axis.

## Crystal Data

$C_7H_{10}O_2Te$, $M = 253.8$, Monoclinic, $a = 23.53(3)$, $b = 4.85(1)$, $c = 16.10(2)$ Å, $\beta = 111.5(1)°$, $V = 1709.5$ Å$^3$, $D_m = 1.98$, $Z = 8$, $D_c = 1.97$, $F(000) = 960$. Space group $B2_1/c$ ($C^5_{2h}$, No.14, in a non-standard setting) from systematic absences: $hk\ell$ when $h + \ell \neq 2n$, $h0\ell$ when $\ell \neq 2n$, and $0k0$ when $k \neq 2n$. Mo-$K_\alpha$ radiation ($\lambda = 0.7107$ Å; $\mu = 31.8$ cm$^{-1}$).

## Structure Determination

The structure was solved by conventional Patterson and Fourier techniques. Scattering factors used were those for the neutral atoms [39], with corrections for the effects of anomalous dispersion [40] ($\Delta f'$,$\Delta f''$) applied to those for tellurium. Initial refinement by full-matrix least-squares methods was with layer scale factors refined separately and all atoms vibrating isotropically. Refinement was continued with the layer scale factors held constant [41] and with the introduction of anisotropic thermal parameters, for all non-hydrogen atoms, of the form $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}\ell^2c^{*2} + 2U_{23}k\ell b^*c^* + 2U_{13}h\ell a^*c^* + 2U_{12}hka^*b^*)]$. All non-methyl hydrogen atoms of the structure were successfully located, at ca. 1 Å from their respective carbon atoms, and were included in the refinement as invariants. The methyl groups were atoms, U was fixed at 0.05 Å$^2$.

In the final stages of refinement the weighting scheme $w = [\sigma^2(F_o) + (0.02|F_o|)^2]^{-1}$ was found appropriate, giving a satisfactory analysis of the variation of $w\Delta^2$ with increasing $\sin\theta/\lambda$ and increasing fractions of $|F_o|$. At convergence no parameter shift was > 0.001σ and a final difference-Fourier map showed no significant features. The final R was 0.044 and R' $\{=[\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{\frac{1}{2}}\}$ was 0.052. A table of observed and calculated structure factors is available from the authors. Most of the calculations were carried out on the ICL 1906A at the University of Birmingham Computer Centre using SHELX-76 [43] with BONDLA and LSQPL being used from XRAY-72 [44] on the CDC 7600 at the University of Manchester Regional Computer Centre. Parameters determined are listed in Tables 1-4; Figure 1 shows a projection of the unit-cell.

## Discussion

The molecules of the structure (Figure 1) are associated with one another by tellurium-tellurium contacts [Te...Te 4.042(5) Å] that form zig-zag polymeric chains of tellurium atoms (Figure 2) about the $2_1$ axes at $x = \frac{1}{4}$ or $\frac{3}{4}$ and $z = 0$ or $\frac{1}{2}$.

The mode of attachment displayed by the β-diketone in the present structure, and in those of (I-IV) [15-18], is new and appears to be unique to tellurium and selenium chemistry. The tellurium atom has a formal oxidation state of +2 and the ligand is therefore bivalent. The tellurium(II) atom binds to the two α-carbon atoms [C(1) and C(5)] of the ligand at distances of 2.202(8) and 2.157(8) Å; these distances are the same, within experimental error, as those found in (I-IV). The α-carbon atom C(1) bonds to H(11), which is axial, and to the equatorial methyl group C(6). The other α-carbon C(5) bonds to two hydrogens [H(51) and H(52)], the former being axial. Both α-carbon atoms bond to the two carbonyl carbons [C(2) and C(4)]. The C-O carbonyl distances [1.22(1), 1.20(1) Å] are both typical of a ketonic carbonyl [9-11]. The γ-carbon atom C(3) is bonded

## Table 1

Fractional atomic coordinates (x $10^5$ for Te; x $10^4$ for C, O; x $10^3$ for H) with estimated standard deviations in parentheses

| Atom | $x$ | $y$ | $z$ |
|---|---|---|---|
| Te | 78183(2) | 5077(11) | 10765(3) |
| C(1) | 8779(3) | 748(15) | 1192(4) |
| C(2) | 9113(3) | −734(16) | 2040(4) |
| C(3) | 9171(3) | 645(16) | 2915(5) |
| C(4) | 8542(3) | 765(16) | 2999(4) |
| C(5) | 8074(3) | 2619(16) | 2340(5) |
| C(6) | 8855(5) | −477(21) | 355(6) |
| C(7) | 9629(4) | −743(20) | 3725(6) |
| O(1) | 9331(2) | −3021(14) | 2050(4) |
| O(2) | 8419(3) | −620(12) | 3529(4) |
| H(11)[a] | 882 | 311 | 126 |
| H(31)[a] | 941 | 250 | 292 |
| H(51)[a] | 826 | 429 | 209 |
| H(52)[a] | 776 | 282 | 227 |
| H(61)[b] | 872(1) | −262(2) | 32(1) |
| H(62)[b] | 927(1) | −30(2) | 23(1) |
| H(63)[b] | 850(1) | 69(2) | −14(1) |
| H(71)[b] | 947(1) | −279(2) | 379(1) |
| H(72)[b] | 967(1) | 45(2) | 431(1) |
| H(73)[b] | 7(1)[c] | −87(2) | 366(1) |

[a] Parameters not refined

[b] Refined as rigid groups pivoting on C(6) and C(7)

[c] Add one

In the non-standard setting of the space group the general equivalent positions are:

$x, y, z$; $-x, -y, -z$; $x, \frac{1}{2}-y, \frac{1}{2}+z$; $-x, \frac{1}{2}+y, \frac{1}{2}-z$;

$\frac{1}{2}+x, y, \frac{1}{2}+z$; $\frac{1}{2}-x, -y, \frac{1}{2}-z$; $\frac{1}{2}+x, \frac{1}{2}-y, z$; $\frac{1}{2}-x, \frac{1}{2}+y, -z$.

methyl group C(7). The resulting six membered heterocyclic ring has the chair conformation (Table 4), a similar result being found in (I–IV). The

The closest intermolecular non-hydrogen atom contacts of the structure are given in Table 3. That between Te...O($2^{III}$) [3.200(1) Å] must represent a non-bonding contact while those between C(1)...O($1^{IV}$), C(5)...O($2^{III}$), C(6)...O($2^{V}$), and C(7)...O($1^{VI}$) [3.38, 3.41, 3.33, 3.39 Å] are all rather long to be considered as possible hydrogen bonding interactions [45-47].

There are two simple ways of describing the hybridization of the tellurium atoms in these compounds; either $\underline{sp}^2$ or $\underline{sp}^3$, in a similar manner to that for tin(II) [48]. In the former, the tellurium atom is considered to have a trigonal planar arrangement, with two bonds to carbon and a lone pair of electrons in the equatorial plane. The two other non-bonding valence electrons will occupy the $\underline{p}$-orbital perpendicular to the trigonal plane. For the $\underline{sp}^3$ case the environment is derived from a tetrahedron with two positions occupied by non-bonding electron pairs. The C-Te-C angle in the present compound [88.4(3)°] is considerably less than either of the ideal values of 120° or 109°28' and can be explained on VSEPR theory [49] as resulting from lone pair repulsion.

## Table 2

Anisotropic thermal parameters (x $10^4$ $Å^2$), with estimated standard deviations in parentheses. $\underline{U}$ was fixed at 0.05 $Å^2$ for all hydrogen atoms.

| Atom | $\underline{U}_{11}$ | $\underline{U}_{22}$ | $\underline{U}_{33}$ | $\underline{U}_{23}$ | $\underline{U}_{13}$ | $\underline{U}_{12}$ |
|---|---|---|---|---|---|---|
| Te | 480(3) | 443(5) | 389(3) | -65(2) | 119(2) | -76(2) |
| C(1) | 493(33) | 409(18) | 329(28) | -12(31) | 192(26) | -37(34) |
| C(2) | 329(28) | 377(17) | 429(30) | -3(32) | 187(25) | 10(32) |
| C(3) | 401(31) | 401(17) | 401(30) | -11(32) | 131(27) | -76(33) |
| C(4) | 496(34) | 413(18) | 332(29) | -50(32) | 147(28) | -32(35) |
| C(5) | 464(35) | 448(20) | 436(33) | -16(32) | 158(30) | 195(34) |
| C(6) | 789(50) | 684(29) | 580(43) | -171(45) | 398(41) | -72(50) |
| C(7) | 576(44) | 586(23) | 595(43) | 170(44) | 142(38) | 24(44) |
| O(1) | 525(27) | 594(16) | 673(30) | 32(31) | 244(25) | -145(30) |

## Table 3

Interatomic distances (Å) and angles (°), with estimated standard deviations in parentheses. Values for rigid groups are not listed.

(a) Distances

| | | | |
|---|---|---|---|
| Te...Te$^{I}$ | 4.042(5) | C(3) - H(31) | 1.06 |
| Te - C(1) | 2.202(8) | C(4) - C(5) | 1.51(1) |
| Te - C(5) | 2.157(8) | C(4) - O(2) | 1.20(1) |
| C(1) - C(2) | 1.487(9) | C(5) - H(51) | 1.07 |
| C(1) - C(6) | 1.54(1) | C(5) - H(52) | 0.70 |
| C(1) - H(11) | 1.15 | Te...O(2$^{III}$) | 3.200(1) |
| C(2) - C(3) | 1.52(1) | C(1)...O(1$^{IV}$) | 3.38(1) |
| C(2) - O(1) | 1.22(1) | C(5)...O(2$^{III}$) | 3.41(1) |
| C(3) - C(4) | 1.54(1) | C(6)...O(2$^{V}$) | 3.33(1) |
| C(3) - C(7) | 1.51(1) | C(7)...O(1$^{VI}$) | 3.39(1) |

(b) Angles

| | | | |
|---|---|---|---|
| Te$^{I}$ - Te - Te$^{II}$ | 73.7(1) | C(2) - C(3) - H(31) | 104 |
| C(1) - Te - C(5) | 88.4(3) | C(4) - C(3) - C(7) | 110.2(7) |
| Te - C(1) - C(2) | 103.4(5) | C(4) - C(3) - H(31) | 120 |
| Te - C(1) - C(6) | 110.5(5) | C(7) - C(3) - H(31) | 99 |
| Te - C(1) - H(11) | 96 | C(3) - C(4) - C(5) | 115.9(7) |
| C(2) - C(1) - C(6) | 115.1(7) | C(3) - C(4) - O(2) | 122.1(6) |
| C(2) - C(1) - H(11) | 113 | C(5) - C(4) - O(2) | 121.9(7) |
| C(6) - C(1) - H(11) | 116 | C(4) - C(5) - Te | 105.0(5) |
| C(1) - C(2) - C(3) | 118.4(6) | C(4) - C(5) - H(51) | 115 |
| C(1) - C(2) - O(1) | 122.0(7) | C(4) - C(5) - H(52) | 130 |
| C(3) - C(2) - O(1) | 119.6(6) | H(51) - C(5) - H(52) | 113 |
| C(2) - C(3) - C(4) | 109.6(5) | Te - C(5) - H(51) | 91 |
| C(2) - C(3) - C(7) | 113.5(7) | Te - C(5) - H(52) | 90 |

Roman numeral superscripts refer to atoms in the following general equivalent positions:

| | | | |
|---|---|---|---|
| I: | $\frac{3}{2}-x$, $-\frac{1}{2}+y$, $-z$ | IV: | $x$, $1+y$, $z$ |
| II: | $\frac{3}{2}-x$, $\frac{1}{2}+y$, $-z$ | V: | $x$, $-\frac{1}{2}-y$, $-\frac{1}{2}+z$ |
| III: | $\frac{3}{2}-x$, $-y$, $\frac{1}{2}-z$ | VI: | $2-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$ |

## Table 4

Least-squares plane through C(1),C(2),C(4), and C(5). The equation of the plane is $12.67\underline{x} + 3.78\underline{y} + 1.63\underline{z} = 11.60$, where $\underline{x},\underline{y},\underline{z}$ are the fractional coordinates of the defining atoms in the direct cell. Atomic deviations from the plane are in Å.

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Te | -1.33 , | C(1) | 0.00 , | C(2) | 0.00 , | C(3) | 0.74 , |
| C(4) | 0.00 , | C(5) | 0.00 , | C(6) | -0.50 , | C(7) | 0.93 , |
| O(1) | -0.59 , | O(2) | -0.59 , | H(11) | 0.95 , | H(31) | 1.74 , |
| H(51) | 0.83 , | H(52) | -0.33. | | | | |

Donaldson and Silver [32-35] have suggested that the colours of p-block materials can be explained in terms of donation of non-bonding valence electron density into bands formed by the overlap of empty p- or d-orbitals on the ligands or on the electron donating atoms themselves. If the $\underline{sp}^2$ hybridization scheme is used, the p-orbital on each tellurium(II) atom will lie approximately along the [010] direction and these are then aligned, together with empty tellurium 4d-orbitals, so that a weak overall bonding orbital is then possible between them. These weak associations would then account for the bright yellow colour of the compound.

When smaller tellurium-tellurium distances are present as in $(C_6H_5)_2Te$ where the Te...Te contacts are alternately 2.7 and 4.2 Å , the colour is red [50]. In the case of $C_4H_8OTeI_2$ there are Te-I...Te distances of 2.8 and 3.7 Å and the colour is again red [29]; if other associations are present, in addition, as in $C_4H_8STeI_2$ where along with Te-I...Te interactions there are I...I interactions, the colour is violet [30]. These colours indicate that the energy of the band edges becomes progressively lower as either the atoms forming them come closer together, or as larger halogens are introduced, as in the case of iodine which has more electrons available for donation into 'bands' [35]. More support for these arguments contains Te-Br...Te alternate associations of 2.65 and 3.59 Å connected by Te...S associations of 3.58 Å. Thus, when I is replaced by Br, the band moves to higher energy, i.e. the energy of orbitals on the parent halide is higher. When the structure of the tellurium compound contains isolated molecules it is colourless, as in $(\underline{p}\text{-}CH_3C_6H_4)_2Te$ [36], or pale yellow, as in phenoxatellurine where the Te...Te distance is 6.04 Å [27]. Tellurium-tellurium distances of ca. 3.9 Å are within the order of associations found for Br...Br in $CsSnBr_3$ and Sn...Sn in SnO [51,52].

In the present structure, the Te...Te distance [4.042(5) Å] is similar to those in (II-IV) (4.068 and 3.95, 3.97, 4.18 and 4.138 Å) [16-18] and is shorter than in (I) (5.05 Å) [15]. The colours of compounds (II-V) are a stronger yellow than that of (I). More evidence for electron density in the Te...Te associations is seen by the larger C-Te-C angle in this compound (88.4°) and in (II-IV) (89.7, 90.8, 89.3°) compared to that of (I) (86.4°), which has weaker associations, showing that lone pair repulsion is reduced in (II-V), indicating less electron density remaining in the lone pairs.

The geometry of the one-dimensional polymer of tellurium atoms in the present structure (Figure 2) is very similar to those found in (II) [16] and (IV) [18]. That in (III) [17], whilst being a one-dimensional polymer aligned along the major growth direction of the crystals with Te...Te distances around 4 Å, is more complicated in its make-up. The Te...Te distances in (II), (IV) and (V) are 4.068, 4.138 and 4.042; the Te...Te...Te angles are 73.4, 72.9 and 73.7°. Structures (II) and (IV) were found to be isomorphous (space group $\underline{P}2_1$), and pseudo-isostructural, with the zig-zag chains of tellurium atoms lying along $2_1$ axes. The effect of the extra equatorial methyl group, substituted on the γ-carbon in (IV), merely served to force the polymers further apart than they were in (II).

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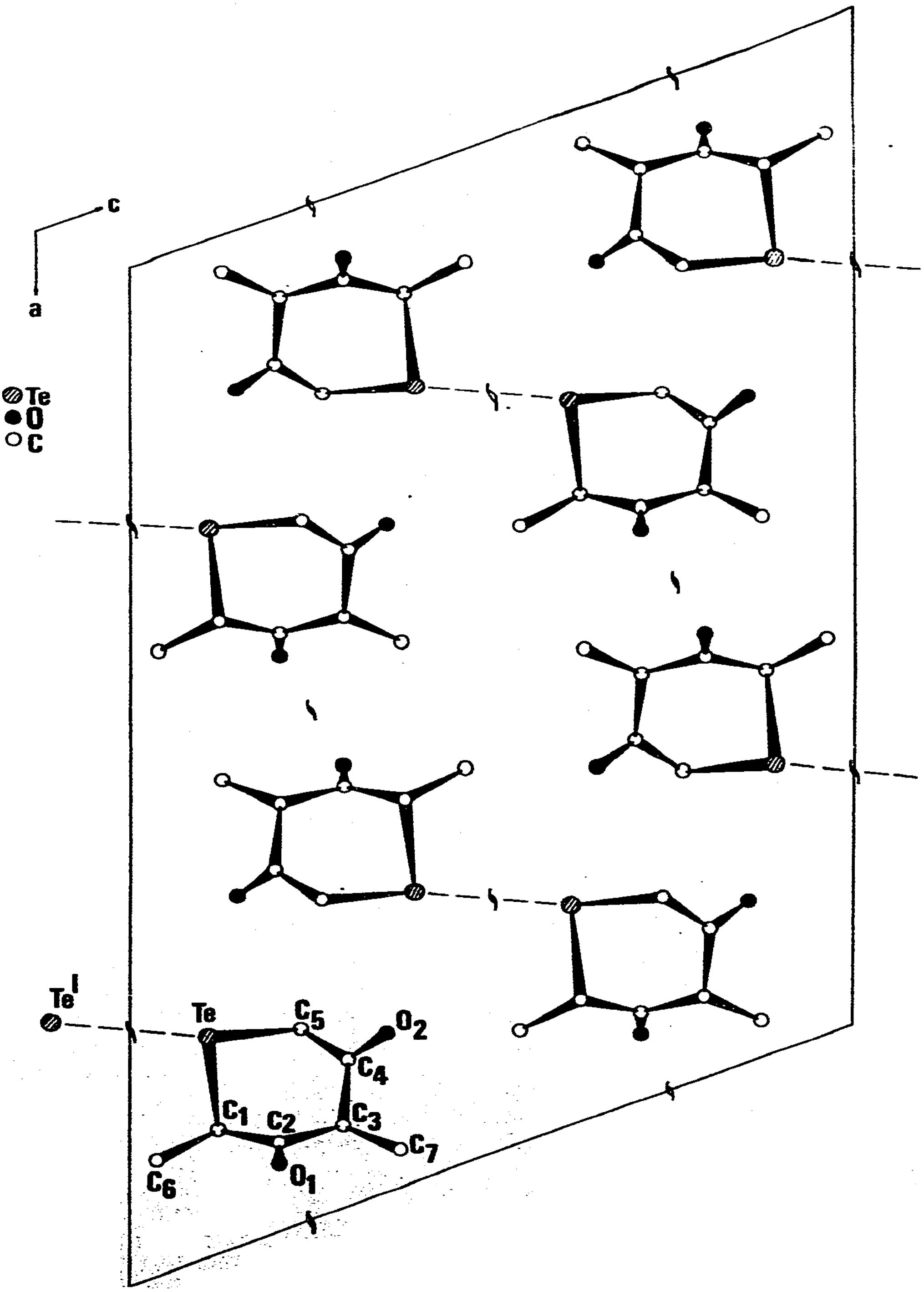
Figure 1. Projection of the structure down [010]. Dashed lines indicate the zig-zag polymers of tellurium atoms lying along the [010] direction. Hydrogen atoms are not shown; however, H(ij) is attached to carbon C(i). H(52) is


c
a
Te
O
C
Te$^I$
Te
$C_5$
$O_2$
$C_4$
$C_1$
$C_2$
$C_3$
$C_7$
$C_6$
$O_1$

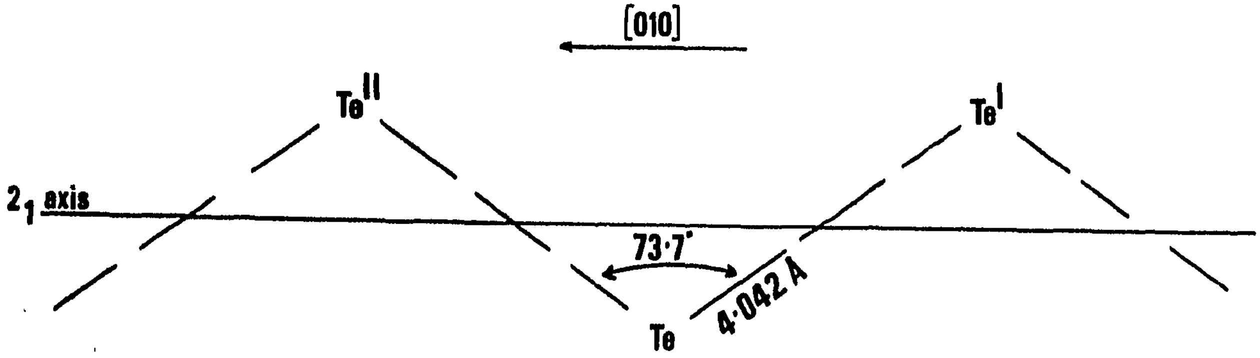


**Figure 2.** Diagrammatic representation of the zig-zag polymeric chain of tellurium atoms.

It was suggested [18] that axial substitution of the methyl group in (IV) would have hindered the ability of the molecules to form these one-dimensional polymers, and it is interesting to note than in (I), where the γ-carbon has both an axial and equatorial methyl group, the Te...Te distance is 5.05 Å and there is no polymeric chain of tellurium atoms, presumably as a result of the axial methyl group on the γ-carbon rendering production of such an arrangement impossible. In the present structure, the equatorial methyl group on one of the α-carbon atoms is replaced by a hydrogen atom. This does not disrupt the nature of the polymer of tellurium atoms but does affect the way these chains of molecules pack together which is seen by the adoption of a different space group, *i.e.* $B2_1/c$ (a non-standard setting of $P2_1/c$). Again the polymers lie along $2_1$ axes. We believe that having observed the same polymer in three separate crystal structure determinations of similar compounds indicates that the major factor controlling the packing of compounds (II), (IV) and (V) is these Te...Te associations and that this adds weight to evidence for the existence of such associations and also to the band theory of the colour properties of these materials [32-35].

Another feature of structures (II), (IV) and (V) that is worthy of comment is that during the formation of the complex in solution each separate methyl group in each compound presumably has a choice of going either axial or equatorial. The fact that in all three structures all the methyl groups are equatorial may be interpreted in that either this observation gives some clue to the kinetics of formation of these complexes or, more likely, that a combination of products is produced and that which crystallizes more readily from the reaction mixture is the isomer that has the ability to form the tellurium polymers, *i.e.* those with no methyl substituents in axial positions.

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