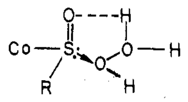
substitution on $H_3O_2^+$). This could occur through an activated complex configuration such as



wherein internal hydrogen bonding to the sulfenato oxygen atom would make OH_2 a poorer leaving group and lower the overall efficacy of $H_3O_2^+$ as a substrate for nucleophilic attack. This hypothesis of a special role for the proton in the H_2O_2–$[(en)_2Co(S(O)CH_2CH_2NH_2)]^{2+}$ reaction is supported by an additional piece of negative evidence. For a wide variety of nucleophilic substrates that obey the general rate law $B = a + b[H^+]$ (eq 4), Hoffmann and Edwards[47] have shown that a plot of log b vs. log a is linear with a slope of 0.95. This implies a similar transition state (i.e., nucleophilic attack on the O–O bond) for both the a and b paths. However, the a and b terms for the H_2O_2–$[(en)_2Co(S(O)CH_2CH_2NH_2)]^{2+}$ reaction do *not* fit on this linear free-energy plot, supporting the view that within the protonated transition state the proton has some definite interaction with the sulfenato moiety.

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Registry No. $[(en)_2Co(S(O)CH_2CH_2NH_2)](ClO_4)_2$, 68645-83-0; $[(en)_2Co(S(O)CH_2CH_2NH_2)](NO_3)_2$, 68645-82-9; $[(en)_2Co(S(O)CH_2CH_2NH_2)](SCN)_2$, 68645-81-8; $[(en)_2Co(S(O)CH_2COO)]ClO_4$, 68645-80-7; $[(en)_2Co(S(O)CH(CH_3)COO)]^+$, 68645-78-3; $[(en)_2Co(S(O)C(CH_3)_2COO)]^+$, 68645-77-2; $[(en)_2Co(S(O)_2CH_2COO)]Cl$, 68698-56-6; $[(en)_2Co(S(O)CH_2CH_2NH_2)](NO_3)(ClO_4)$, 68645-76-1; $[(en)_2Co(S(CO)CH_2COO)]Cl$, 68698-55-5; $[(en)_2Co(SCH_2CH_2NH_2)](ClO_4)_2$, 40330-50-5; $[(en)_2Co(SCH_2COO)]ClO_4$, 26743-67-9; $[(en)_2Co(SCH(CH_3)COO)]^+$, 60828-74-2; $[(en)_2Co(SC(CH_3)_2COO)]ClO_4$, 68645-87-4; $[Co(en)_3]^{2+}$, 23523-25-3.

Supplementary Material Available: Tables A–F giving $|F_o|$ and F_c, anisotropic thermal parameters, positional parameters, bond lengths and bond angles involving hydrogen atoms, and ϵ_{365}^{obsd}–$[H^+]$ data (14 pages). Ordering information is given on any current masthead page.

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(35) $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$; $R_2 = [\sum w(|F_o| - |F_c|)^2/\sum w(F_o)^2]^{1/2}$.
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Crystal and Molecular Structure of Dimethyltellurium Tetraiodide, $(CH_3)_2TeI_4$

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$(CH_3)_2TeI_4$ crystallizes in the space group $P\bar{1}$ with $a = 7.460$ (3) Å, $b = 12.600$ (5) Å, $c = 6.592$ (3) Å, $\alpha = 82.83$ (3)°, $\beta = 103.54$ (3)°, $\gamma = 106.45$ (3)°, $V = 576.6$ Å^3, and $Z = 2$. The crystal structure was determined with diffractometer data and refined to $R_1 = 0.061$ using anisotropic thermal parameters for all atoms. The compound is an adduct of $(CH_3)_2TeI_2$ with I_2 linked by intermolecular I–I bonds and weak Te–I bonds and does not contain Te(VI).

Introduction

Dialkyltellurium tetraiodides are prepared by the reaction of the corresponding dialkyltellurium diiodides with molecular iodine.[1-3] The chemical behavior and the spectra give no evidence for an oxidation state of +6 for tellurium but are in agreement with a weakly bound adduct of R_2TeI_2 with I_2.[4,5] Smith and Thayer suggested an interaction between the central tellurium and the molecular iodine, as the Te–I stretching bands in the IR spectrum do not change going from R_2TeI_2 to R_2TeI_4.[4] But in α–$(CH_3)_2TeI_2$ the tellurium already forms intermolecular Te–I bonds.[6] Additional bonding of the tellurium is not possible without breaking these intermolecular

Table I. Atomic Parameters and Esd's[a]

atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Te	0.722 21 (9)	0.807 29 (5)	0.442 18 (9)	2.65 (3)	2.58 (2)	2.31 (2)	0.62 (2)	0.26 (2)	−0.21 (2)
I(1)	0.376 58 (10)	0.674 94 (7)	0.629 48 (12)	2.98 (3)	3.86 (3)	4.17 (3)	−0.19 (2)	1.33 (3)	−1.47 (3)
I(2)	1.050 12 (12)	0.910 11 (7)	0.280 12 (11)	3.84 (4)	4.23 (3)	3.17 (3)	−0.04 (3)	1.43 (2)	0.03 (2)
I(3)	0.107 88 (11)	0.555 84 (7)	0.172 55 (12)	2.95 (3)	3.64 (3)	4.69 (3)	0.60 (2)	0.89 (3)	−0.56 (3)
I(4)	0.460 50 (10)	0.906 49 (7)	0.890 84 (11)	2.47 (3)	4.32 (3)	3.47 (3)	0.33 (2)	0.07 (2)	−0.81 (2)
C(1)	0.683 9 (19)	0.661 7 (11)	0.284 4 (20)	3.9 (6)	4.3 (6)	5.2 (6)	0.4 (4)	1.7 (5)	2.3 (5)
C(2)	0.895 4 (17)	0.760 2 (11)	0.730 0 (17)	3.4 (5)	4.6 (6)	2.8 (4)	0.3 (4)	−0.2 (4)	0.7 (4)

[a] The form of the anisotropic temperature factor is $\exp[-0.25(a^{*2}h^2B_{11} + \ldots + 2hka^*b^*B_{12} + \ldots)]$.

Table II. Interatomic Distances and Angles (in Å and deg)[a]

Te–I(1)	3.082 (2)	I(3)–I(3)^{III}	2.748 (2)
Te–I(2)	2.809 (2)	I(4)–I(4)^{IV}	2.763 (2)
Te–C(1)	2.14 (1)	I(1)–I(3)	3.456 (2)
Te–C(2)	2.15 (1)	I(1)–I(4)	3.404 (2)
Te–I(2)^{I}	3.957 (2)		
Te–I(4)^{II}	3.938 (2)		
I(1)–Te–I(2)	174.63 (4)	C(2)–Te–I(2)^{I}	76.8 (4)
I(1)–Te–C(1)	86.3 (3)	C(2)–Te–I(4)^{II}	173.3 (3)
I(1)–Te–C(2)	86.0 (3)	I(2)^{I}–Te–I(4)^{II}	101.85 (5)
I(1)–Te–I(2)^{I}	108.53 (4)	Te–I(1)–I(3)	95.75 (5)
I(1)–Te–I(4)^{II}	100.58 (5)	Te–I(1)–I(4)	84.65 (5)
I(2)–Te–C(1)	89.9 (3)	I(3)–I(1)–I(4)	140.45 (4)
I(2)–Te–C(2)	90.7 (3)	Te–I(2)^{I}–Te^{I}	105.24 (4)
I(2)–Te–I(2)^{I}	74.76 (4)	I(1)–I(3)–I(3)^{III}	175.20 (5)
I(2)–Te–I(4)^{II}	82.65 (5)	I(1)–I(4)–I(4)^{IV}	178.23 (5)
C(1)–Te–C(2)	96.8 (5)	Te^{V}–I(4)–I(4)^{IV}	79.26 (6)
C(1)–Te–I(2)^{I}	163.2 (3)	Te^{V}–I(4)–I(1)	99.37 (5)
C(1)–Te–I(4)^{II}	82.6 (4)		

[a] Roman numerals as superscripts refer to the following equivalent positions: I, $2-x$, $2-y$, $1-z$; II, x, y, $z-1$; III, $-x$, $1-y$, $-z$; IV, $1-x$, $2-y$, $2-z$; V, x, y, $1+z$.

bonds and this should have an influence on the Te–I stretching bands. The present study was performed to determine the intermolecular bonding between $(CH_3)_2TeI_2$ and I_2.

Experimental Section

$(CH_3)_2TeI_4$ was prepared by the method of Smith and Thayer.[3] As the compound readily loses iodine, the crystals were sealed in glass capillaries for the X-ray measurements. Lattice parameters were determined from precession films and refined with the indexed lines of a Guinier film (Cu Kα_1 radiation, λ 1.540 51 Å, quartz monochromator, calibration substance $Pb(NO_3)_2$, a = 7.856 (1) Å). Intensity data were collected with a Stoe Weissenberg diffractometer (Mo Kα radiation, λ 0.7107 Å, graphite monochromator). Reflections were measured by an ω scan with a scan rate of 1.2°/min, with background measurements for 25 s on both sides of the scan, and with varying scan ranges, which were calculated by the formula $\Delta\omega = A + B \sin \mu/\tan \theta'$ (where θ' is half the angle between the counter and horizontal plane, μ is the equiinclination angle, and A and B are constants with values around 1.6–2.0° and 1.0–2.0° respectively). The crystal had approximately the dimensions 0.18 × 0.18 × 0.77 mm and the rotation axis coincided with the c axis. A total of 3150 unique reflections of the layers $hk0$–$hk9$ up to θ_{max} = 30° were measured. For 267 reflections σ_I was greater than the intensity I. These reflections got the weight w = 0 and were not included in the refinement. The intensities were corrected for absorption (T_{min} = 0.156, T_{max} = 0.240).

Crystal data: $(CH_3)_2TeI_4$, M_r = 665.27, triclinic, space group $P\bar{1}$, a = 7.460 (3) Å, b = 12.600 (5) Å, c = 6.592 (3) Å, α = 82.83 (3)°, β = 103.54 (3)°, γ = 106.45 (3)°, V = 576.57 Å^3, Z = 2, d_{calcd} = 3.832 g cm^{-3}, μ(Mo Kα) = 131.2 cm^{-1}.

Structure Determination. The heavy atoms could be located in a Patterson synthesis and the carbon atoms were found by a Fourier synthesis. Least-squares refinement with anisotropic thermal parameters for all atoms and individual scale factors for all layers gave R_1 = 0.073 and R_2 = 0.089 (w was set equal to $1/\sigma_F^2$).[7] An analysis of the F_o and F_c values showed great discrepancies for some very strong reflections; 24 of the most affected reflections were excluded from the further refinement (w = 0) leading to R_1 = 0.061 and R_2 = 0.067 for reflections with $w \neq 0$ and R_1 = 0.078 for all reflections. An attempt to refine the structure in the space group $P1$ did not give better results.

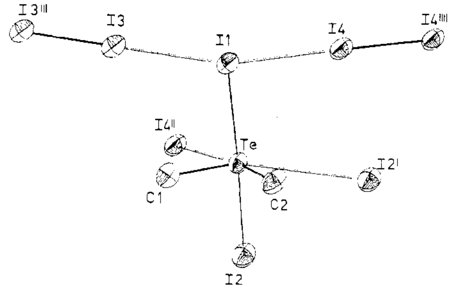

Figure 1. Molecular structure and labeling of $(CH_3)_2TeI_4$. The coordinates of the positions I, II, III, and IV are given in Table II.

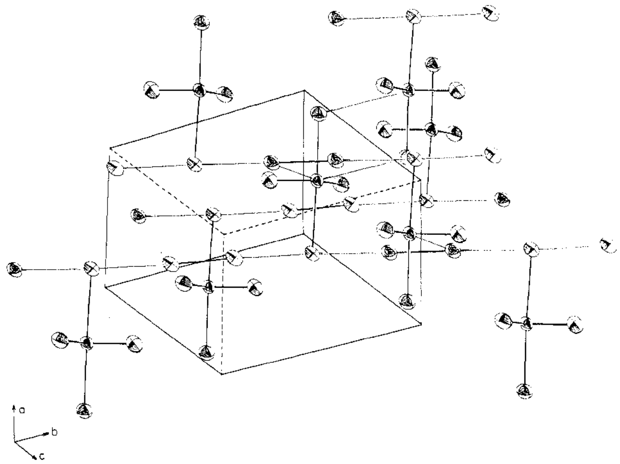

Figure 2. View of the cell of $(CH_3)_2TeI_4$ showing the intermolecular arrangement.

Final parameters are listed in Table I. Scattering factors by Cromer and Mann were used,[8] and the dispersion corrections for Te and I were included.[9] The interatomic distances and angles are given in Table II.

Structure Description and Discussion

In agreement with the spectroscopic results, the structure is built up by $(CH_3)_2TeI_2$ and I_2 molecules and does not contain Te(VI). The compound should be formulated as dimethyltellurium diiodide–diiodine(1/1). The iodine molecules are on both sides bound to iodine atoms of $(CH_3)_2TeI_2$ molecules (Figures 1 and 2) and not to the central tellurium as proposed by Smith and Thayer.[4] The I_2 molecule and the two other iodine atoms form a linear chain, which is designated as an I_4 group.[10] Only one of the iodine atoms bound to tellurium, I(1), is connected with the I_2 molecules and is part of two crystallographically independent but very similar I_4 groups. The connection of the I_2 molecules via I(1) leads to zigzag chains along the [112] direction. Besides this chain the tellurium forms weak intermolecular bonds, one to I(2)^{I}, which is directly bound to another tellurium, and one to I(4)^{II}, which belongs to one of the bridging iodine molecules. C(1)–Te–I(2)^{I} and C(2)–Te–I(4)^{II} are slightly bent (163 and 173°). These weak intermolecular bonds connect the different zigzag chains.

The coordination around tellurium is formed by two trans Te–I bonds and two Te–C bonds in the plane perpendicular to I(1)–Te–I(2). The Te–C distances have the expected values, while the Te–I distances are longer than the sum of the covalent radii (2.70 Å[11]) but lie in the range found for similar compounds.[12] The C(1)–Te–C(2) angle is somewhat larger than 90°. The coordination around tellurium is completed by the two weak intermolecular Te–I bonds with $I(2)^{I}$ and $I(4)^{II}$ and can be described as a distorted octahedron.

The coordination around tellurium found for this compound can be compared with the known structures of analogous compounds of the formula R_2TeI_2,[12] for which a trigonal-bipyramidal arrangement with the lone electron pair in the equatorial plane is suggested by the VSEPR model.[13] The angle between the ligands in the equatorial plane is expected to be somewhat smaller than 120° and the axial distances are somewhat elongated. All structures generally show this arrangement with equatorial angles ranging from 91 to 101° and axial distances from 2.83 to 3.10 Å. In nearly all structures there are intermolecular Te–I bonds, which complete a distorted octahedral coordination around tellurium.

Another possible description of the bonding of tellurium can be given by considering only s and p orbitals. The ^{125}Te Mössbauer spectra of R_2TeX_2 compounds can be rationalized by a bonding scheme involving 5p orbitals with some 5s character: the linear X–Te–X bond is formed by the $5p_z$ orbital of tellurium and two σ orbitals of the halogens with four electrons (3c–4e bond), and for the bonds in the equatorial plane the $5p_x$ and $5p_y$ orbitals with some 5s admixture are used.[14]

This is a possible explanation for the elongation of the axial Te–I bonds (compared with the sum of the covalent radii) and for the weak intermolecular Te–I bonds. The 3c–4e bonds are weaker than the corresponding 2c–2e bonds. The two bonds in the linear group I–Te–I are strongly correlated: the lengthening of one Te–I bond results in a shortening of the other. Intermolecular bonds of the outer iodine atoms decrease the strength of the intramolecular bond. The distance from tellurium to I(1) which is connected with two I_2 molecules is much longer than that to I(2) which has only one weak intermolecular contact. This effect is observed in the other R_2TeI_2 structures, too.

The weak intermolecular Te–I bonds are formed by the interaction of the C–Te bond with a lone electron pair of the iodine. The approximately linear structure supports this view. The bonding may be described as a very unsymmetrical 3c–4e bond or as a secondary bond.[15]

I_2 bridges between two partly negatively charged iodine atoms were found in some other structures, too.[10] The four atoms form an almost linear group and the angles at the outer iodine atoms, which often are part of another molecule, deviate only slightly from 90°. The inner distance is somewhat longer than in an isolated iodine molecule. The outer distance is relatively long but significantly shorter than the sum of the van der Walls radii. The distances and angles found for $(CH_3)_2TeI_4$ are in agreement with those of other known structures.

The bonding scheme used for I_3^- can be extended to this group. The 5p orbitals of the atoms along the molecular axis form a four-center bond occupied by six electrons. The found distances are qualitatively in agreement with this model. The 5p orbitals perpendicular to the molecular axis are used for the bonding of the outer iodine atoms with atoms of other molecules and the angles around I(1) in this compound should have values around 90°. The Te–I(1)–I values deviate only slightly from this value, while the angle I(3)–I(1)–I(4) is much greater. The corresponding angles in some other structures with similar zigzag chains vary from 100 to 176.[16] There is no obvious reason for it and an explanation cannot be given. The steric hindrance between iodine atoms of the same chain does not forbid an angle of 90°.

Registry No. $(CH_3)_2TeI_4$, 41235-23-8; $(CH_3)_2TeI_2{\cdot}I_2$, 52585-06-5.

Supplementary Material Available: A listing of structure factor amplitudes (14 pages). Ordering information is given on any current masthead page.

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