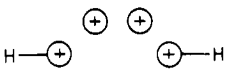
C and the most unstable type (19 a″) exhibits the maximum number of nodes D. Only the empty ligand orbital A and the filled ligand orbital B appreciably interact with cobalt d_{xz} and d_{yz} orbitals, respectively (Table VI and coordinate system as defined in Figure 5). Ligand types A and B, correspond to the 9 a′ and 6 a″ orbitals of Figure 5.



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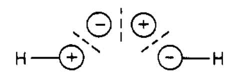

D

A comparison of the interactions between the isoelectronic (η^5-C_5H_5)Co and Fe(CO)$_3$ fragments with π orbitals of an HN_4H fragment is provided in Table VII. Although general π-orbital splittings and their compositions are similar for the two complexes, the percent tetraazadiene π^* (type A) character differs for 28 a′ and 31 a′ in the cobalt and iron complexes, respectively. Greater contribution from the ligand π^* orbital in the cobalt case leads to increased back-bonding compared with Fe(CO)$_3$(HN_4H). The iron d_{z^2} character found in 31 a′ is also noteworthy since this orbital cannot effectively overlap with the tetraazadiene π^* orbital. This could explain the nearly fivefold increase in intensity of the metallacycle $\pi \rightarrow \pi^*$ transition (vide infra) in the cobalt complexes, since cobalt employs d_{xz} orbital character in 28 a′.

Transition-state calculations[43] place the lowest one-electron transition of (η^5-C_5H_5)Co(HN_4H) at 2.38 eV, and the electronic spectrum of (η^5-C_5H_5)Co($CH_3N_4CH_3$) exhibits a weak feature (Figure 4) at 598 nm (2.07 eV). The weak intensity arises from lack of overlap between the d_{z^2} orbital, 29 a′, and the π^* orbitals of the N_4 ring. Although three other transitions, 17 a″, 28 a′, and 27 a′ → 30 a′ should lie at slightly higher energies, only two other transitions are apparent (Figure 4). We attribute the most intense band at 424 nm to the $\pi \rightarrow \pi^*$ excitation localized on the metallacycle (28 a′ → 30 a′). Calculations may incorrectly estimate the energy of 17 a″ → 30 a′, or this transition could be hidden beneath the intense 424-nm feature. Just as for the iron(0) tetraazadiene complex,[2a,b] the vivid colors of the cobalt(I) tetraazadiene compounds may be attributed to the low-lying unoccupied metallacycle π^* orbital.

Acknowledgment. We thank the National Science Foundation for support of this work (Grants CHE 78-01615 and 81-05069 to W.C.T. and CHE 80-09671 to J.A.I.).

Registry No. IVa, 80738-16-5; IVb, 76418-81-0; IVc·$^1/_2C_6H_6$, 80738-17-6; IVd, 80738-18-7; IVe, 80738-19-8; (η^5-C_5H_5)Co(HN_4H), 80738-20-1.

Supplementary Material Available: Tables of the positional and thermal parameters, of the root-mean-square amplitudes of vibration, and a listing of observed and calculated structure amplitudes (22 pages). Ordering information is given on any current masthead page.

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5,6:11,12-Bis(ditelluro)tetracene: Synthesis, Molecular, and Supramolecular Properties[1]

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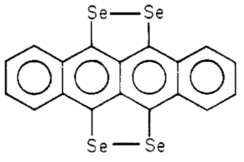
Received December 2, 1981

5,6:11,12-Bis(ditelluro)tetracene (5,6,11,12-tetratellurotetracene, TTeT), synthesized in 13% yield from 5,6,11,12-tetrachlorotetracene and a new sodium ditelluride reagent, absorbs at longer wavelengths in both solution and solid state and is oxidized electrochemically at a lower potential, compared to its selenium analogue. The structure of TTeT, a monoclinic crystal (a = 11.746 (4) Å, b = 4.364 (2) Å, c = 15.831 (5) Å, β = 90.57°, space group $P2_1/n$, R = 0.030, R_w = 0.037), exhibits short interstack contacts of 3.701 (1) Å.

The organoselenium π donors which give ion-radical solids with metallic states below 30 K are 5,6:11,12-bis(diseleno)tetracene 1 (TSeT)[2] and derivatives of tetraselenafulvalene (TSeF, 2), and ambient pressure superconductivity has been observed to date only in the 2:1 perchlorate salt of the tetramethyl derivative of **2**.[3] Substitution of tellurium for selenium in 1 and 2 is expected



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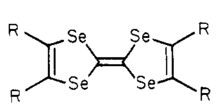

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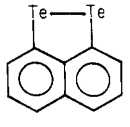

4

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† On sabbatical leave at GTE Laboratories, 1980–1981, from the Department of Chemistry, Eastern Nazarene College, Quincy, MA 02170. This work was supported in part by National Science Foundation Grant SP1-8160202.

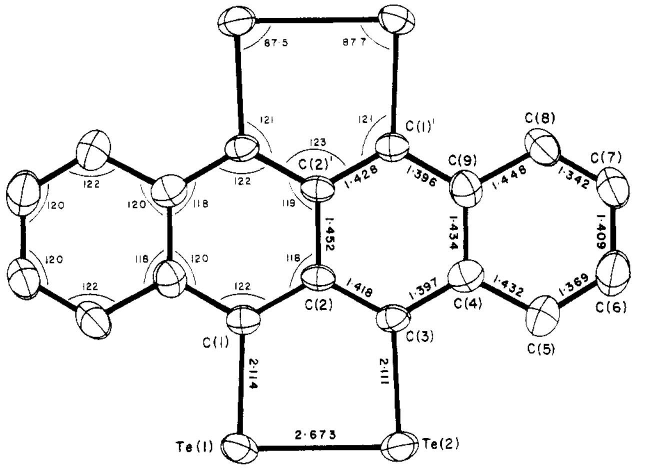
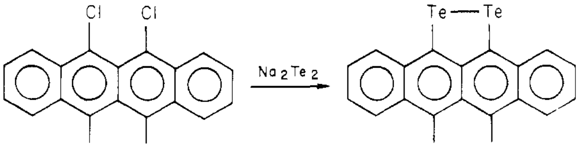


Figure 1. The molecular structure of $C_{18}H_8Te_4$, showing 50% probability ellipsoids for atoms refined anisotropically. The molecule occupies a crystallographic center of symmetry (midpoint of the C(2)–C(2)′ bond). Standard deviations of the bond lengths and angles are Te–Te = 0.001 Å, Te–C = 0.007 Å, C–C = 0.009–0.013 Å, Te–Te–C = 0.2°, Te–C–C = 0.5°, and C–C–C = 0.6–0.8°. The molecule is planar with no deviations >0.016 (7) Å.

to enhance both the molecular polarizability[4] and the intermolecular contacts involving the chalcogen in ion-radical solids involving these donors. The availability of the tellurium compounds has been anticipated,[5] and we now report the synthesis of tetratellurotetracene (3, TTeT), its crystal and molecular structure, and several molecular and solid-state properties pursuant to our interest in peri-dichalcogenide derivatives of aromatic hydrocarbons.[6]

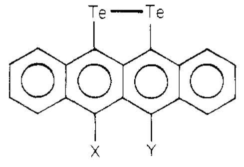
An important development relative to the present work was the synthesis and characterization of naphthalene 1,8-ditelluride (4).[7] Relative to its lighter chalcogen analogues, 4 absorbed at longer wavelengths and was more easily oxidized electrochemically[7b] even though all of the naphthalene 1,8-dichalcogenides have vertical ionization energies of ca. 7.1 eV.[6,7c]

We synthesized TTeT by reaction of tetrachlorotetracene[8] (5) with a new sodium ditelluride reagent formed directly from the elements in a 1:1 atomic ratio in a dipolar aprotic solvent such as dimethylformamide (DMF) or hexamethylphosphoramide.[9]



5 3

In a representative procedure, a magnetically stirred suspension of Te powder (7.65 g, 0.06 mol) and sodium (1.38 g, 0.06 mol) in DMF (100 mL) under argon was heated at 110 °C for 1 h. After the mixture was cooled to 55 °C, solid 5 (5.0 g, 0.0137 mol) was added followed by DMF (100 mL). This mixture was heated at 45–55 °C for 20 h when it was poured into salt water. The solid precipitate was dried and extracted with acetone and benzene to remove ditellurotetracenes 6.[10] The residue was crystallized from chlorobenzene to give TTeT as a black solid, 1.28 g (13% yield), mp >360 °C. Anal. Calcd for $C_{18}H_8Te_4$: C, 29.43; H, 1.10; Te, 69.47. Found: C, 29.24; H, 1.15; Te, 69.58. Our initial identification of the black solid as TTeT followed from its mass spectrum which exhibited the pattern expected for a four Te ion with the most intense peak at *m/e* 736, relative abundance 10.9%; a major fragment observed corresponds to loss of one Te atom.



6a, X = Y = H
b, X = Y = Cl
c, X = H, Y = Cl

In chlorobenzene solution, we observed the following absorption spectrum for TTeT (λ_{max} 763 nm (log ϵ 4.16), 695 (3.97), 465 (3.73), 338 (4.92)). The long wavelength absorption reveals a significant red shift compared to TSeT[12] (λ_{max} 716 nm, (log ϵ 4.1), 661 (4.0), 471 (3.9)). The solid-state spectrum of TTeT, observed by diffuse reflectance, exhibits a broad maximum in the remission function at 810 nm, red shifted compared to its solution spectrum and also the diffuse reflectance of TSeT (λ_{max} 760 and 640 nm).

We observed surface adsorption complications with both TTeT and TSeT, possibly due to crystallization of an ion-radical solid, while attempting a comparative electrochemical study. In chlorobenzene solution 0.10 M in tetra-*n*-butylammonium tetrafluoroborate at a sweep rate of 100 mV/s, TTeT exhibits an anodic peak at +0.22 V while the peak for TSeT is observed at +0.32 V, both measured relative to the Ag/AgCl electrode in saturated NaCl. These data suggest that TTeT is more easily oxidized in solution than its selenium counterpart, a situation analogous to the naphthalene 1,8-dichalcogenides.[7b] Reversible electrochemical behavior was reported for tetra-

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(1) (a) A preliminary account of portions of this work was presented at the International Conference on Low-Dimensional Conductors, Boulder, CO, August 9–14, 1981: Sandman, D. J.; Stark, J. C.; Hamill, G. P.; Burke, W. A.; Foxman, B. M., paper 3A-24. (b) Portions of this work will also be presented at the National Meeting of the American Chemical Society, Las Vegas, NE, March 29–April 2, 1982, paper ORGN 71.

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(8) Balodis, K. A.; Livdane, A. D.; Medne, R. S.; Neiland, O. Y. *J. Org. Chem. USSR (Engl. Transl.)* **1979**, *15*, 343. Our samples of 5 exhibit a melting point of 221–222 °C vs. 212–214 °C reported by Balodis et al. Anal. Calcd for $C_{18}H_8Cl_4$:C, 59.06; H, 2.20; Cl, 38.74. Found: C, 59.28; H, 2.28; Cl, 38.7.

(9) The synthesis of TSeT from 5 and sodium diselenide in DMF has been reported.[8]

(10) These compounds have not been isolated in sufficient quantity to allow a complete characterization; they were identified from mass spectra. The visible absorption maxima of 6a–c are respectively 610, 642, and 623 nm. These compounds are red-shifted vs. dithiotetracene whose visible maximum is at 561 nm.[11] The isolation of 6a–c as well as the mass spectrometric detection of di- and trichlorotetracene, indicates the reducing power of the Na_2Te_2 reagent. Irreversible reduction of 5 is revealed by a cathodic peak at −0.71 V (vs. Ag/AgCl in saturated NaCl) in DMF using 0.1 M tetra-*n*-butylammonium tetrafluoroborate.

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(12) Marschalk, C. *Bull. Soc. Chim. Fr.* **1952**; 800.

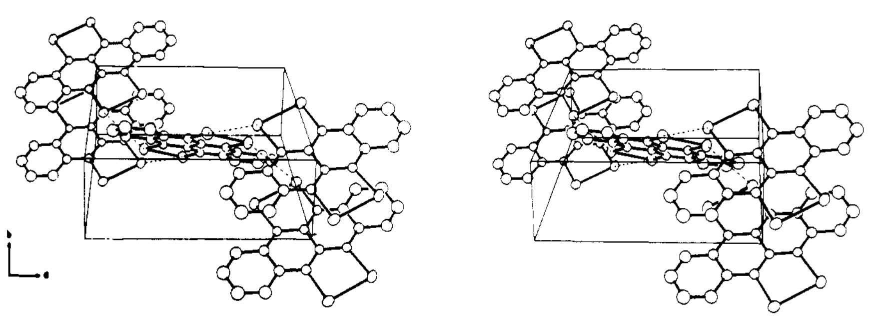

Figure 2. A stereoview of the packing of $C_{18}H_8Te_4$, showing (i) stacks of TTeT molecules along the *b* axis and (ii) short interstack Te–Te contacts of 3.701Å (dashed lines), which connect stacks along [101].

thiotetracene (TTT) in CH_2Cl_2 solution.[13]

Our samples of TTeT revealed no evidence of decomposition in the course of X-ray photographic studies of single crystals kept in air over a 3-month period. While derivatives of tetrathiafulvalene often reveal the formation of *S*-oxides in mass spectrometry and ESCA studies,[14] our mass spectral data (vide supra) have not revealed peaks at values of m/e higher than the molecular ion. ESCA studies of our samples of TTeT reveal two peaks in both the Te($3d_{3/2}$) and Te($3d_{5/2}$) levels at binding energies of 584.3 and 586.3 eV and 573.8 and 575.9 eV, respectively, as well as an O(1s) peak, suggesting surface oxidation.[15] After argon sputtering of the sample surface, lines at 583.9 and 573.5 eV remained, values identical with those reported for diphenyl ditelluride.[16]

We determined the crystal and molecular structure of TTeT because such studies of neutral organochalcogen donors are of continuing interest with respect to binding forces[17] in these solids and the use of molecular geometry to infer degree of charge-transfer complexes derived from these donors.[18] TTeT crystallizes in the monoclinic space group $P2_1/n$, with a = 11.746 (4) Å, b = 4.364 (2) Å, c = 15.831 (5) Å, β = 90.57 (5)°, Z = 2, ρ_{calcd} = 3.01 g cm^{-3}, and ρ_{obsd} = 3.01 (2) g cm^{-3}. This crystal is isomorphous with a polymorph of TSeT.[1a] Full-matrix least-squares refinement of positional and anisotropic thermal parameters for Te and C atoms, with H atoms included as fixed contributions (r_{C-H} = 0.95 Å), using 1283 data for which $F > 3.92\sigma(F)$ and $2\theta_{MoK\alpha} \leq 53°$, gave R = 0.030 and R_w = 0.037.[19] Subsequent to our preliminary report,[1a] we learned of another structural study of TTeT.[20] The two structure determinations are in agreement except for a significant difference in cell constants[21] and the degree of precision of the analyses.[22] The study of Shibaeva and Kaminskii[20] was carried out to slightly higher resolution ($2\theta_{MoK\alpha} \leq 55.4°$), but only 38% as much data (482 reflections for which $I > 1.96\sigma$) were obtained, despite the fact that the crystal size was ca. 4.5 times that used in the present study. It is probable that the cell constant and diffraction-quality differences between the two experiments are a comment on the probable purity and/or crystal quality of the materials. A complete report of experimental detail, coordinates, and bond lengths and angles for the present study is available as supplementary material.

Table I. Observed and Theoretical Distances (Å) for Tetratellurotetracene and Tetracene

bond	$C_{18}H_8Te_4$ [a]	$C_{18}H_{12}$ [b]	
C(5)-C(6)	1.356 (14)[c]	1.364	1.358
C(6)-C(7)	1.409 (12)	1.429	1.440
C(4)-C(5)	1.440 (8)[c]	1.436	1.445
C(4)-C(9)	1.434 (10)	1.421	1.422
C(3)-C(4)	1.397 (7)[c]	1.390	1.385
C(1)-C(2)	1.423 (7)[c]	1.412	1.415
(C2)-C(2)′	1.452 (13)	1.419	1.413

[a] Present work. [b] Dewar, M. J. S.; Gleicher, G. J. *J. Am. Chem. Soc.* **1965**, *87*, 685. The bond lengths in the two columns were calculated by the PPP and SPO treatments, respectively. [c] Values of averaged pairs of bond lengths assuming D_{2h} symmetry; standard deviations are the larger of the standard deviation of the mean or one-half the difference between the pair of averaged values.

Figure 1 shows the molecular structure of TTeT. The observed C–C bond lengths are in close agreement with theoretical calculations on tetracene (Table I) as well as with the structures of tetracene and TTT.[23] The packing of the molecules is shown in Figure 2. The plane-to-plane intrastack distance is 3.732 Å; further, there are rather short *interstack* Te(1)–Te(2)′ contacts of 3.701 Å, slightly longer than the value of 3.630 Å reported for $C_{16}H_{12}Te_2$.[24] Thus, the stacks are interconnected via infinite ...Te(1)–Te(2)...Te(1)′–Te(2)′... chains, with a Te(1)–Te(2)···Te(1)′ angle of 77.1° and a Te(2)···Te(1)′–Te(2)′ angle of 116.9°. There are infinite, equal Te(1)–Te(1)′–Te(1)″... contacts along a chain of 4.055 Å. The Te–Te and Te–C distances are near the values observed for diphenyl ditelluride (2.712 (2) and 2.115 (16) Å, respectively), and the average Te–

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(21) Cell constant determinations on the Syntex $P2_1$ diffractometer at Brandeis University (T = 21 (1) °C) have been calibrated by using a 0.15-mm spherical ruby crystal obtained from the National Research Council of Canada. A reviewer has pointed out that, given thermal expansion coefficients of (2–4) × 10^4/°C, the cell constants in ref 20 may not differ significantly from those reported in the present work. This of course would require that the temperature of measurement for the former determination be ~30 °C.

(22) Using a relationship derived by Cruickshank: Cruickshank, D. W. J. in "Computing Methods in Crystallography"; Rollett, J. S., Ed.; Pergamon Press: Oxford, England, 1965, p 116, we noted that our standard deviations should be ~28% of those observed in ref 20. Upon inspection of that work, we discovered that the standard deviations (calculated by using the values for coordinates in Tables I and II of ref 20) were in error and should be: Te–Te = 0.003 Å, Te–C = 0.020 Å, C–C = 0.034–0.044 Å, C9–C9′ = 0.066 Å. The latter esd is for the C–C bond in which the atoms are related by a center of symmetry.

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Te–C angle, 87.62 (14)°, is considerably smaller than that found in the unconstrained Ph_2Te_2 molecule (98.9 (15)°).[25]

Acknowledgment. The work at Brandeis University was supported in part by the Office of Naval Research. We thank D. Dugger, F. X. Pink, and M. A. McGrath for providing mass spectral, ESCA, and diffuse reflectance data, respectively, G. D. Zoski for the use of electrochemical apparatus and helpful discussions, W. A. Burke and S. Meyler for general technical assistance, and Joanne McLaughlin for typing the manuscript.

Registry No. 3, 64479-92-1; **5**, 62409-66-9.

Supplementary Material Available: Tables of (i) experimental data, (ii) atomic coordinates, (iii) anisotropic thermal parameters, (iv) bond lengths and angles, and (v) observed and calculated structure factors (10 pages). Ordering information is given on any current masthead page.

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Palladium-Catalyzed Reactions of Glycals with (1,3-Dimethyl-2,4(1*H*,3*H*)-pyrimidinedion-5-yl)mercuric Acetate. Facile Regio- and Stereospecific *C*-Nucleoside Syntheses

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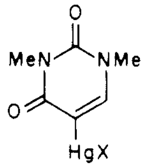
Received September 9, 1981

Reactions of the glycals, 3,4,6-tri-*O*-acetyl-D-glucal, 3,4-di-*O*-acetyl-D-arabinal, and 3,4-di-*O*-acetyl-D-xylal, with (1,3-dimethyl-2,4(1*H*,3*H*)-pyrimidinedion-5-yl)mercuric acetate in the presence of a stoichiometric quantity of $Li_2Pd(OAc)_2Cl_2$ resulted in regio- and stereospecific carbon–carbon bond formation between C-5 of the pyrimidine moiety and C-1 of the carbohydrate. Regiospecificity in this reaction results from the dipolar nature of the enol ether double bond; stereospecificity derives from exclusive approach of the metallopyrimidine reagent to the face of the carbohydrate (enol ether) opposite that bearing the allylic acetate group.

In a preliminary report,[2] we described palladium-catalyzed reactions of glycals, 1,2-unsaturated carbohydrates, with a pyrimidin-5-ylmercuric salt which result in regiospecific carbon-carbon bond formation between C-5 of the pyrimidine moiety and C-1 of the carbohydrate. This reaction constitutes a new, facile *C*-nucleoside[3] synthesis. We have also reported related studies involving palladium-catalyzed reactions of other enol ethers and acetates.[4-6] Recently Czernecki and Gruy[7] have reported related palladium-catalyzed glycal arylation reactions.[8]

In the present report we (a) describe further studies of the palladium-catalyzed reactions of glycals which we have found to be sensitive to the nature of the anions present in the reaction mixture,[9] (b) discuss mechanistic aspects of the addition reaction (which determines product regio- and stereochemistry) and of adduct decomposition reactions (which give rise to three discrete *C*-nucleoside products),[10] (c) discuss the chromatographic and spectrometric properties of these product *C*-nucleosides, and (d) describe cyclization of an acyclic *C*-nucleoside produced in the primary reaction.



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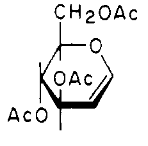

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Regio- and Stereospecific Reactions of a Pyrimidinylpalladium Reagent with Glycals. Reaction of (1,3-dimethyl-2,4(1*H*,3*H*)-pyrimidinedion-5-yl)mercuric acetate[4] (**1**, X = OAc) with 3,4,6-tri-*O*-acetyl-D-glucal[11] (**2**) in acetonitrile in the presence of a stoichiometric quantity of the palladium(II) salt $Li_2Pd(OAc)_2Cl_2$ afforded one major product, (*Z*)-1,2-dideoxy-1-(1,2,3,4-tetrahydro-1,3-dimethyl-2,4-dioxo-5-pyrimidinyl)-D-*arabino*-hex-1-enitol 3,4,6-triacetate (**3**) and two minor ones, 5-(4,6-di-*O*-acetyl-2,3-dideoxy-α-D-*erythro*-hex-2-enopyranosyl)-1,3-

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[1] (a) Faculty of Science, Lebanese University, Beirut, Lebanon: Visiting Professor of Chemistry and Fulbright Scholar at Oregon Graduate Center, 1980. (b) Department of Chemistry, Lehigh University, Bethlehem, PA 18018; to whom reprint requests should be addressed.

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