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5,6:11,12-BIS(DITELLURO) TETRACENE: SYNTHESIS, MOLECULAR AND SU--ETC(U)
JAN 82 D J SANDMAN, J C STARK, B M FOXMAN N00014-76-C-0A22
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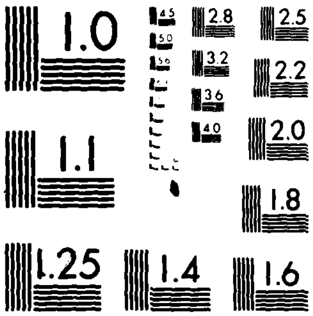

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5,6,:11,12-Bis(diterlluro)tetracene (TTeT), synthesized in 13% yield from 5,6, 11,12-tetrachlorotetracene and a new sodium ditelluride reagent, absorbs at longer wavelengths in both solution and solid state and is oxidized electrochemically at a lower 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, $\beta = 90.57°$, space group $P2_1/n$, $R = 0.030$, $R_w = 0.037$), exhibits short interstack contacts of 3.701(1)A.

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TECHNICAL REPORT NO. 9

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

by

Daniel J. Sandman, James C. Stark and Bruce M. Foxman

Prepared for Publication

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Brandeis University

Department of Chemistry

Waltham, Massachusetts 02254

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Tetratellurotetracene: Synthesis, Molecular, and Supramolecular Properties[1]

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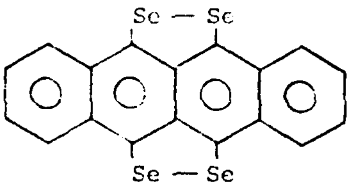
Abstract

Tetratellurotetracene (TTeT), synthesized in 13% yield from 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 analog. The structure of TTeT, a monoclinic crystal, a = 11.74[illegible](4), b = 4.364(2), c = [illegible].831(5) Å; β = 96.57°, space group $I2_1/a$, R = 0.036, R_w = 0.[illegible]37, exhibits short interstack contacts of [illegible].7[illegible]1(1) Å.

[+] On sabbatical leave at GTE Laboratories, 1980-81. This work was supported in part by National Science Foundation Grant SPI-8160202. Permanent address: Department of Chemistry, Eastern Nazarene College, Quincy, MA 02170.

The organoselenium pi-donors which give ion-radical solids with metallic states below 30°K are tetraselenotetracene (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 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].



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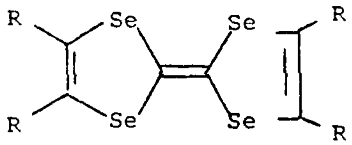

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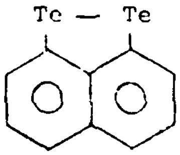

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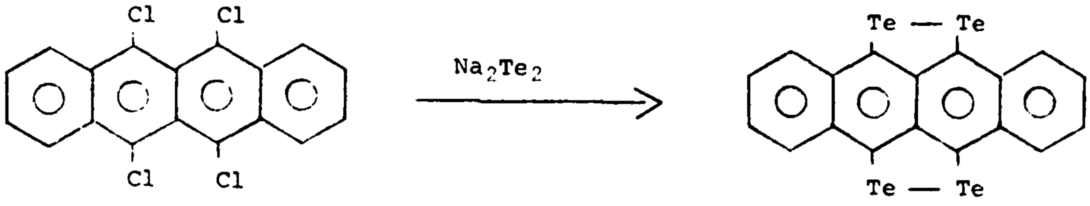
An important development relative to the present work was the synthesis and characterization of naphthalene-1,8-ditelluride (4)[7]. Relative to its lighter chalcogen analogs, 4 absorbed at longer wavelengths and was more easily oxidized electrochemically[7b] even though all of the naphthalene-1,8-dichalcogenides 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]



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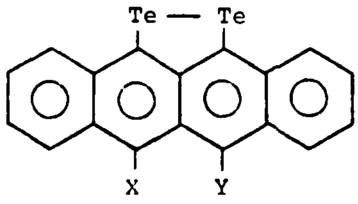
In a representative procedure, a magnetically stirred suspension of Te powder (7.65 g, 0.06 gm atom) and sodium (1.38 g, 0.06 gm atom) in DMF (100 ml) under argon was heated at 110° for an hour. After cooling to 55°, solid 5 (5.0 g, 0.0137 mole) was added followed by DMF (100 ml). This mixture was heated at 45-55° for 20 hr 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°. Anal. Calcd for $C_{18}H_8Te_4$: C, 29.45; 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.

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).



6 a, x=y=H
b, x=y=Cl
c, x=H, y=Cl

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_{mzx} 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/sec, 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. This data suggests 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 tetrathiotetracene (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 three 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 to those reported for diphenylditelluride[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 $\underline{a}$=11.746(4), $\underline{b}$=4.364(2), $\underline{c}$=15.831(5)Å; β=90.57(5)$^\circ$; Z=2; ρ_{calc}=3.01 g-cm^{-3}; ρ_{obs}=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}=0.95Å), using 1283 data for which $F>3.92\sigma(F)$ and $2\theta_{MoK\alpha} \leq 53^\circ$, 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 ($2\theta_{MoK\alpha} \leq 55.4^\circ$), 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, bond lengths and angles for the present study is available as supplementary material.

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 3.732Å; further, there are rather short *interstack* Te(1)-Te(2)' contacts of 3.701Å, slightly longer than the value of

TABLE I

OBSERVED AND THEORETICAL DISTANCES 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.

3.630 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 4.055Å. The Te-Te and Te-C distances are near the values observed for diphenylditelluride (2.712(2) and 2.115(16), respectively) and the average Te-Te-C angle, 87.62(14)° is considerably smaller than that found in the unconstrained Ph_2Te_2 molecule (98.9(15)°).[25]

Acknowledgements The work at Brandeis University was supported in part by the Office of Naval Research. The authors 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.

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 (*n* pages). Ordering information is given on any current masthead page.

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9. The synthesis of TSeT from 5 and a reagent formed from sodium and selenium in a 1:1 atomic 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 versus 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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21. Cell constant determinations on the Syntex $P2_1$ diffractometer at Brandeis University (T = 21(1)° C) have been calibrated using a 0.15 mm spherical ruby crystal obtained from the National Research Council of Canada. A referee has pointed out that, given thermal expansion coefficients of $2 - 4 \times 10^{4}$/°C, the cell constants in reference 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 reference 20. Upon inspection of that work, we discovered that the standard deviations (calculated using the values for coordinates in Tables 1 & 2 of ref. 20) were in error and should be: Te-Te, 0.003; Te-C, 0.020; C-C, 0.034-0.044; C9-C9', 0.066Å. The latter esd is for the C-C bond in which the atoms are related by a center of symmetry.

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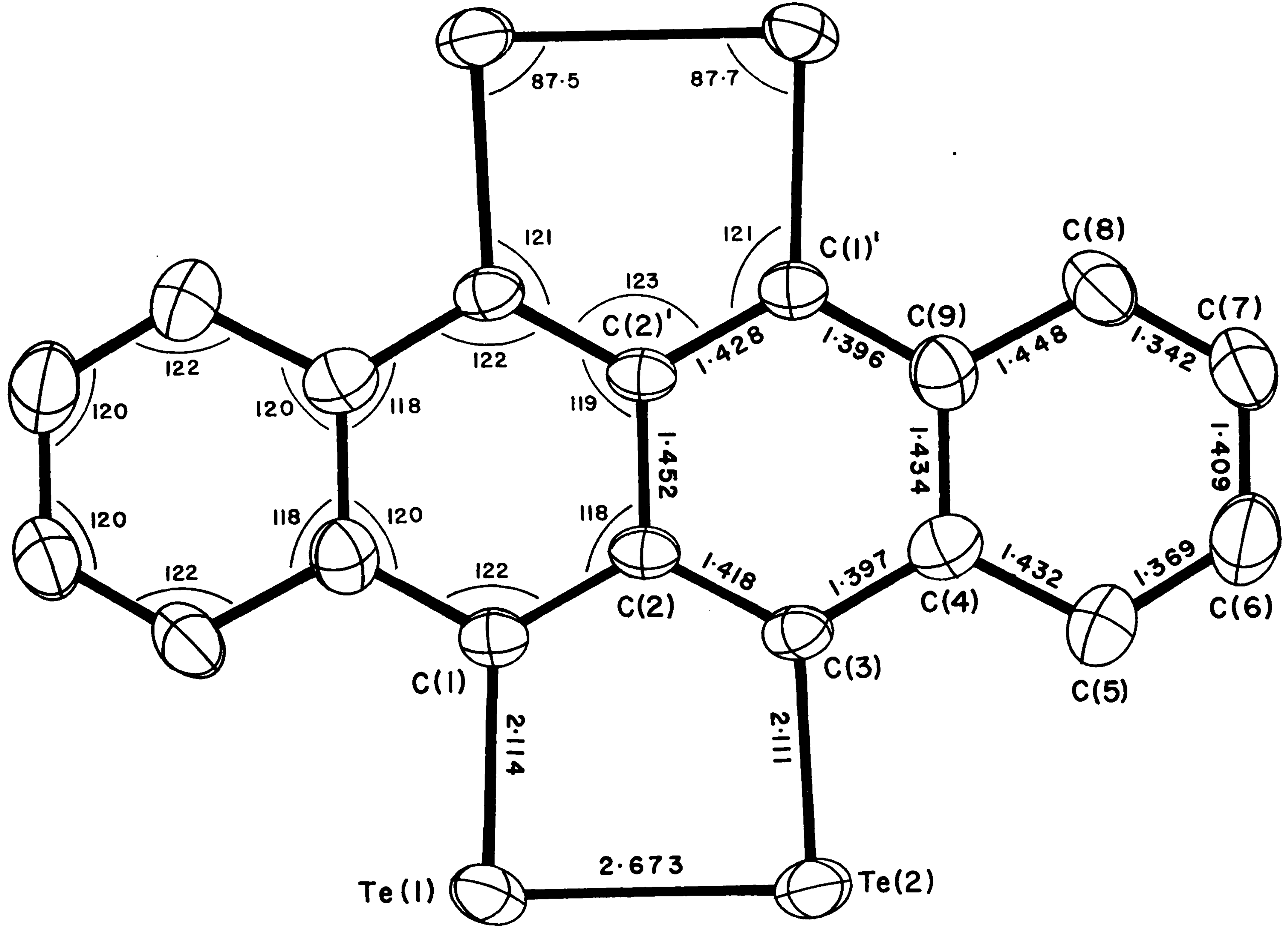
FIGURE CAPTIONS

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 (mid-point of the C(2)-C(2)' bond). Standard deviations of the bond lengths and angles are: Te-Te, 0.001Å; Te-C, 0.007Å; C-C, 0.009-0.013Å; Te-Te-C, 0.2 ; Te-C-C, 0.5 ; C-C-C, 0.6-0.8 . The molecule is planar with no deviations > 0.016(7)Å.

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].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1·434
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1·418
1·397
1·432
1·369
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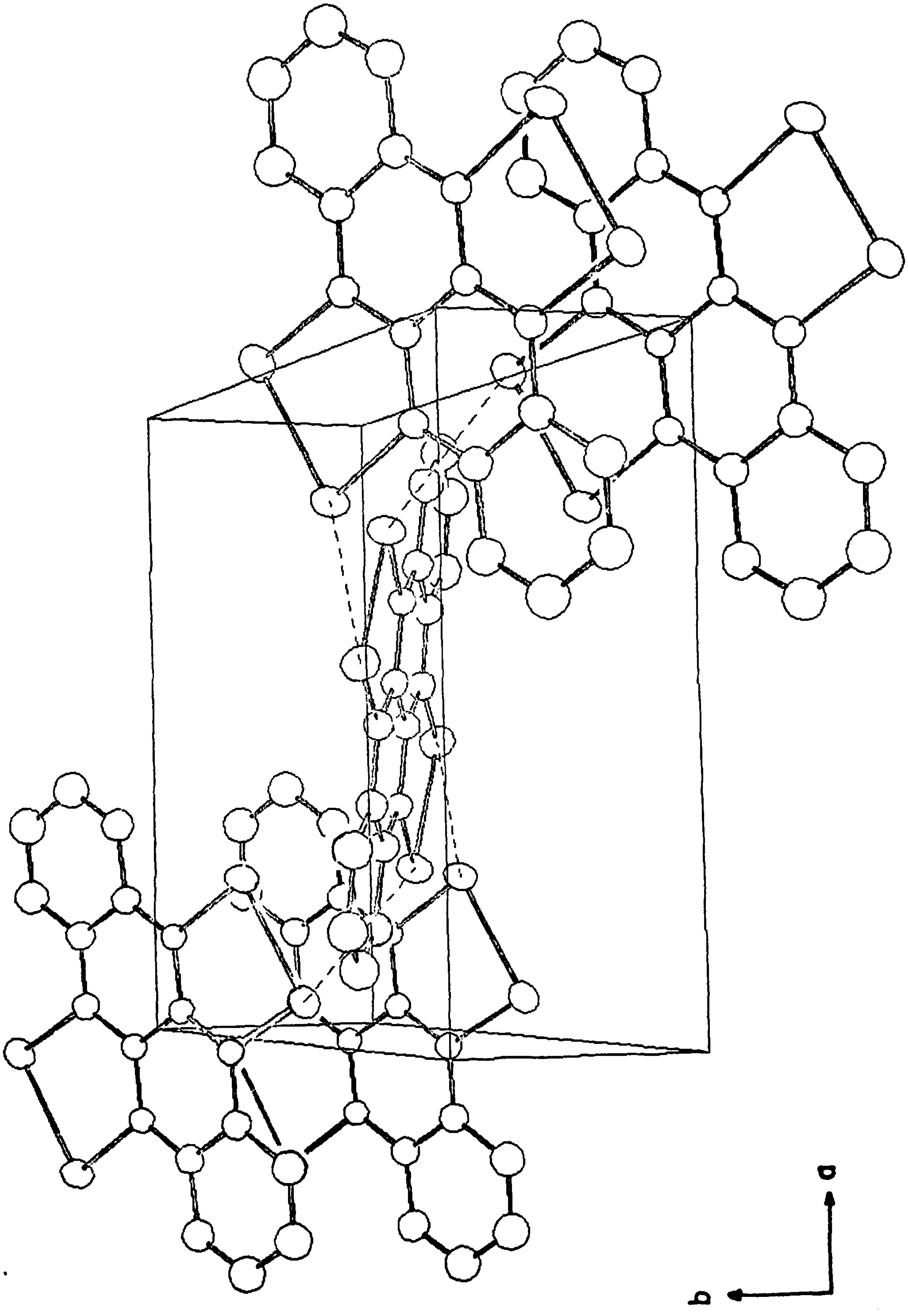
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