have confirmed this structure and have shown that a water molecule completes a distorted octahedral arrangement around the Te atom.

As a final example we present results for Me$_3$TeI (Figure 2f). Unfortunately, it is not possible to extract accurate anisotropic shielding information from the powder pattern, which has a width at half-height of ~10 kHz. Application of magic angle spinning reduces the width to ~4 kHz. The spectral width is probably dominated by unresolved dipolar and *J* coupling to one or more spin-$^5/_2$ ^{127}I nuclei. Complications from this kind of interaction may prove to be a problem in NMR studies of many heavy nuclides, especially because high coordination numbers are favored.

However, we believe that these examples have shown the utility of solid-state ^{125}Te NMR in deriving structural information. Especially the possibility of observing resolved *J* couplings suggests further studies of this nature.

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Tetratellurafulvalene (TTeF)

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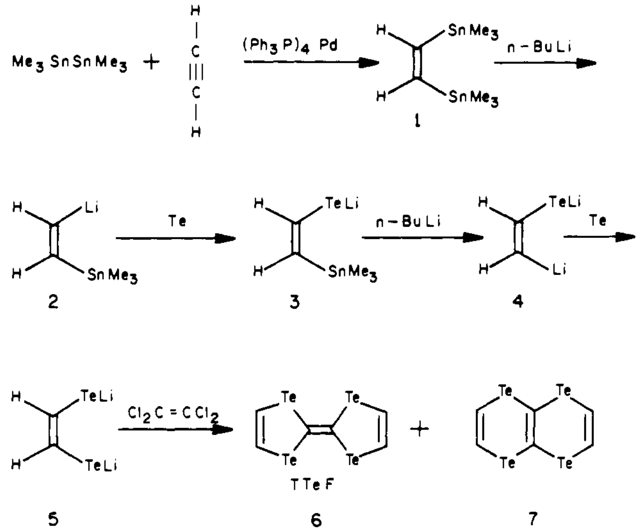
One of the long-standing goals of organic solid-state research has been the creation of new heterocyclic π-donors in an attempt to alter the electrical conductivity of radical cation salts derived from these donors.[1] Reported herein is the first synthesis of tetratellurafulvalene (TTeF, **6**). This compound completes the interesting series starting with tetrathiafulvalene (TTF), first prepared by Wudl[2] and Coffen,[3] and followed 4 years later by the selenium analogue, tetraselenafulvalene (TSF), synthesized by Engler.[4]

The electrical properties of salts derived from this class of compounds display a rich variety of behavior, ranging from insulators[5] (all DA stack salts and those with unit charge transfer) to anisotropic metals[6] (for example, tetracyanoquinodimethane salts of TTF, TSF, and tetramethyltetraselenafulvalene, TMTSF) and superconductors[7] (for example, (TMTSF)$_2$ClO$_4$ and related salts and bis(ethylenedithio)tetrathiafulvalene (BEDT-TTF) salts, (BEDT-TTF)$_2$I$_3$).

Scheme I



Three important trends can be expected upon the replacement of S or Se with Te atoms in these complex salts. The more diffuse p and d orbitals centered on tellurium should give larger conduction bandwidths due to increased intrastack interactions and result in materials with reduced electron scattering and enhanced metallic electrical conductivity. In addition this increase in orbital spatial extension ought to increase the interchain interactions giving rise to a more two- or three-dimensional character. This extended dimensionality should help suppress the various instabilities which often lead to insulating ground states in quasi-one-dimensional organic conductors. Finally, the greater polarizability of tellurium should reduce the on-site Coulombic repulsion and help support doubly charged species.[8] Unless the molecular component can support doubly charged species, only a correlated type of conductivity is possible.

The synthesis of TTeF adds to a small but growing list of tellurium heterocyclic π-donors recently prepared.[9] With the preparation of this compound, it should be possible not only to compare the sulfur, selenium, and tellurium series of compounds but also to construct binary and ternary alloys, for example, (TTeF)$_x$(TSF)$_y$(TTF)$_{1-x-y}$TCNQ. In addition, it may be possible to use the route described below to prepare selenium compounds like TSF.

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Table I. Cyclic Voltammetry Data[a]

donor[b]	$E_{1/2}(1)$, V	$E_{1/2}(2)$, V	$\Delta E_{1/2}$, V
TMTTF	0.36	0.84	0.48
TMTSF	0.55	0.94	0.39
HMTSF	0.56	0.93	0.37
TTF	0.47	0.81	0.34
TSF[c]	0.62	0.90	0.28
TTeF	0.59	0.84	0.25

[a] Volts vs. SCE at Pt button electrode; 0.2 M tetrabutylammonium tetrafluoroborate in CH_2Cl_2; 200 mV/s sweep rate. [b] TMTTF = tetramethyltetrathiafulvalene; TMTSF = tetramethyltetraselenafulvalene; HMTSF = hexamethylenetetraselenafulvalene; TTF = tetrathiafulvalene; TSF = tetraselenafulvalene. [c] Calculated from data reported by: Engler, E. M.; Kaufman, F. B.; Green, D. C.; Klots, C. E.; Compton, R. N. *J. Am. Chem. Soc.* **1975**, *97*, 2921.

TTeF was prepared in 36% yield utilizing a synthetic methodology that should be useful in producing a variety of new tetratellurafulvalenes (Scheme I). Acetylene was bubbled through a THF solution of hexamethylditin containing a catalytic amount of tetrakis(triphenylphosphine)palladium to give (*Z*)-1,2-bis(trimethylstannyl)ethylene, according to a literature procedure by Mitchell et al.[10] (unfortunately, this reaction is applicable only to terminal alkynes or tetramethyltetratellurafulvalene (TMTTeF) could have been easily made as well).[11] The purified distannylalkene (**1**) was then treated with1.1 equiv of *n*-BuLi at −78 °C (1 h); then 0.90 equiv of elemental tellurium was added and the reaction mixture was warmed to −20 °C. After all of the tellurium had completed reacting, the first two steps were repeated. The reaction mixture was then cooled to −78 °C, whereupon 0.5 equiv of tetrachloroethylene was added. An immediate reaction took place as evidenced by a temperature increase and a rapid color change. The reaction mixture was then allowed to slowly warm up to 25 °C over an 18-h period. After filtration of the reaction mixture, the CS_2 extractable solids were isolated to give a 36% (unoptimized) yield. This yield is somewhat lower than that of some other previously synthesized tetratellurafulvalenes (e.g., BDMT-TTeF, 75%[9f]). This can be accounted for due to the equilibrium that exists in the first transmetalation reaction. The extent of this equilibrium was determined by treating compound **1** with 1 equiv of alkyllithium and trapping **2** with D_2O. The ratio of the expected product to the starting material, **1** was determined to be 1:1–2:1 by NMR spectroscopy.

Chromatography (silica, CS_2) followed by spectroscopic study proved the compound to be the heterofulvalene TTeF.[12] The mass spectrum of TTeF exhibits a molecular ion M^+ (m/z 588, relative intensity 70%) with an isotope pattern closely resembling a computer simulation. Other major cluster fragments (>25% relative intensity) are 562 M − C_2H_2 (28%), 306 $C_4H_2Te_2$ (50%), 282 $C_2H_2Te_2$ (100%), 256 Te_2 (60%), and 130 Te (45%). High-resolution mass spectrometry shows peaks in the molecular ion cluster at 595.6573 (calcd for $C_6H_4{}^{130}Te_4{}^+$, 595.6562) and in the M − C_2H_2 cluster at 569.6439 (calcd for $C_4H_2{}^{130}Te_4{}^+$, 569.6406) and a peak corresponding to $C_6H_4{}^{130}Te_3{}^{128}Te^+$ at 593.6570 (calcd 593.6545).

The electrochemical data, obtained by cyclic voltammetry, is shown in Table I. TTeF, as expected, shows two reversible one-electron oxidations. The value for the first oxidation potential ($E_{1/2}(1)$) indicates an ionization potential for TTeF between TTF and TSF. With an ionization potential in this range it should be possible to form organic metals from TTeF. The other expected electrochemical result is that the $\Delta E_{1/2}$ follows the trend TTeF < TSF < TTF, suggesting that the intramolecular Coulomb repulsion energy decreases in this series. It is interesting to note that the $\Delta E_{1/2}$ for BEDT-TTF is 0.24 V, making TTeF at 0.25 V one of the better donors in this respect.[8b]

The occurrence of an irreversible oxidation peak at 1.15 V in the crude TTeF suggests that the mixture contains some of the six-membered ring isomer **7**. This assignment is based on the similar electrochemical behavior of hexamethylenetetratellurafulvalene (HMTTeF) and its six-membered ring isomer, as well as the crystallographic structure of this compound.[13,14] Tetratellurafulvalene and **7** can be separated by HPLC and the components identified by using a stopped flow–UV scan method.[15] We estimate that the reaction product contains about 85% TTeF and about 15% **7**. A similar ratio was obtained by comparing anodic peak currents in the cyclic voltammogram.

While we are currently attempting to obtain single crystals of a variety of charge-transfer complexes and radical cation salts of TTeF, we believe it is desirable to make this synthesis available to the chemistry and physics community. This will ensure that the broadest range of solid-state properties is explored.

Acknowledgment. This work was supported by the National Science Foundation—Solid State Chemistry Program Grant DMR-8615305. We thank Dr. Joseph L. Kachinski, Jr., for collecting the mass spectral data. Purchase of the mass spectrometer was made possible by the NIH (1-S10-RR02318-01). Purchase of the 400-MHz NMR was made possible by the NIH (1-S10-RR01934) and the NSF (PCM 83-03176).

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Bonding in 1,3-Cyclodisiloxanes: ^{29}Si NMR Coupling Constants in Disilenes and 1,3-Cyclodisiloxanes

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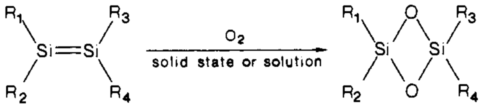
The nature of the bonding in 1,3-cyclodisiloxanes (**6–10**) has been the subject of considerable discussion in the chemical literature since their discovery in 1981.[1] In this class of compounds, the observed silicon–silicon internuclear distances are remarkably short, as determined by X-ray crystallographic studies.[2] In the



1 $R_1 = R_2 = R_3 = R_4 =$ Mes **6**
2 $R_1 = R_2 = R_3 =$ Mes $R_4 =$ Xyl **7**
3 $R_1 = R_2 =$ Mes $R_3 = R_4 =$ Xyl **8**
4 $R_1 =$ Mes $R_2 = R_3 = R_4 =$ Xyl **9**
5 $R_1 = R_2 = R_3 = R_4 =$ Xyl **10**

Mes = 2,4,6-trimethylphenyl, Xyl = 2,6-dimethylphenyl

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