$Cp_2Zr(CH_2Ph)(L)^+$ complexes from **4** were unsuccessful, as the CH_3CN ligand does not undergo substitution by THF (even in refluxing THF, Scheme I) or PMe_2Ph, presumably because the η^2-benzyl ligand effectively blocks the central coordination site of the Cp_2Zr^{IV} fragment.[11] However, the THF complex $[Cp_2Zr(CH_2Ph)(THF)][BPh_4]$ (**2**) was obtained (100% NMR, 54% isolated) as an orange solid containing 0.5THF of crystallization directly from **5** by reaction with $[Cp_2Fe][BPh_4]$[12] in THF (eq 2). Complex **2** contains a normal η^1-benzyl ligand (^1H NMR)

$$\underset{\mathbf{5}}{\mathrm{Cp_2Zr(CH_2Ph)_2}} + \mathrm{[Cp_2Fe][BPh_4]} \xrightarrow{\mathrm{THF}} \underset{\mathbf{2}}{\mathrm{[Cp_2Zr(CH_2Ph)(THF)][BPh_4]}} + \mathrm{Cp_2Fe} + {}^{1}/_{2}\mathrm{PhCH_2CH_2Ph} \qquad (2)$$

and reacts with CH_3CN to yield **4** and with PMe_2Ph to yield **6** via nucleophilic ring opening of the coordinated THF (Scheme I).

^1H NMR spectroscopy reveals that **2** undergoes significant THF dissociation in CH_2Cl_2 to form the η^2-benzyl species **3**, as illustrated in Scheme I. The −50 °C spectrum of a dilute (0.008 M) CD_2Cl_2 solution of **2** includes resonances for **2** (ca. 50%, δ 6.15 (s, 10 H), 3.15 (s, 4 H), 2.69 (s, 2 H), 1.67, (s, 4 H)) as well as resonances assignable to **3** (ca. 50%, δ 5.94 (s, 10 H), 2.88 (s, 2 H)) and resonances for free THF of appropriate intensity. The presence of an ortho H resonance at δ 6.7 (not observed for **2** in THF-d_8) integrating for 2 H indicates that **3** has an η^2 structure similar to that of **4**.[8] The available data do not establish the extent of CH_2Cl_2 coordination to **3**.[13] Addition of THF shifts the equilibrium in the expected manner.[14] Solutions of **2** in CH_2Cl_2 polymerize ethylene (1 atm, 23 °C); by analogy to the results for the methyl complex **1**, complex **3** may be a key intermediate in this process. To date, efforts to directly probe the interaction of **3** with ethylene have been unsuccessful, as THF exchange between **2** and **3** is fast under polymerization conditions.

The synthesis of **2** (eq 2) apparently involves one-electron oxidation of d^0 **5** which proceeds with concomitant extrusion of benzyl radical. Benzylferrocene, the expected product of coupling of benzyl radical and Cp_2Fe^+, is observed as a minor product.[15] Other $Cp_2Zr(R)_2$ complexes ($R^- = CH_3^-, Ph^-$) are oxidized to cationic complexes in a similar manner.[16] One-electron oxidation of d^n ($n > 0$) complexes (i.e., removal of a primarily nonbonded d electron) promotes a variety of reactions including CO insertion, ligand exchange, reductive elimination, and C–H activation.[17] The present results show that one-electron oxidation of d^0 $Cp_2Zr(R)_2$ compounds provides unique access to highly reactive $Cp_2Zr(R)^+$ complexes. This approach should be applicable to the synthesis of other types of reactive, cationic, d^0 organometallic complexes from neutral precursors containing carbanionic alkyl ligands.

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Supplementary Material Available: Characterization data for **2–6**, summary of X-ray data for **4**, and tables of atomic and thermal parameters and bond distances and angles for **4** (9 pages). Ordering information is given on any current masthead page.

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(11) However, 4 undergoes rapid CH_3CN exchange with free CH_3CN (second-order rate constant ca. 600 M^{-1} s^{-1} at −56 °C), as well as ligand substitution by PMe_3 to produce a labile PMe_3 complex and free CH_3CN. Insoluble $Ag[BPh_4]$ reacts with Zr alkyls only in CH_3CN solvent.

(12) Prepared by addition of $Na[BPh_4]$ to an aqueous $[Cp_2Fe]_2[SO_4]$ solution. Jolly, W. L. *The Synthesis and Characterization of Inorganic Compounds*; Prentice-Hall: Englewood Cliffs, NJ, 1970, p 487.

(13) Complex **2** decomposes (23 °C, 48 h) in CH_2Cl_2 to $Cp_2Zr(CH_2Ph)(Cl)$.

(14) $K_{eq} = 0.008$ M at −50 °C. Exchange of free and coordinated THF is slow at −50 °C ($k < 3$ s^{-1}) but rapid at 30 °C ($k_f = 60$ s^{-1}, $k_r = 7800$ M^{-1} s^{-1}, from coalescence of Cp resonances).

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(16) $[(C_5H_4Me)_2Fe][BPh_4]$ is also effective in these reactions and is more stable than $[Cp_2Fe][BPh_4]$. Efforts to determine if eq 1 involves a similar one-electron oxidation process are under way.

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CP/MAS ^{125}Te NMR in Solids: An Example of ^{125}Te–^{35,37}Cl *J* Coupling[†]

Michael J. Collins and John A. Ripmeester*

Division of Chemistry
National Research Council of Canada
Ottawa, Ontario K1A 0R9, Canada

Jeffery F. Sawyer

Department of Chemistry, University of Toronto
Toronto, Ontario M5S 1A1, Canada

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A great number of publications have appeared in which cross-polarization, dipolar decoupling, and magic angle spinning have been used to obtain NMR spectra of the lighter nuclides such as ^{13}C, ^{15}N, ^{29}Si, and ^{31}P in the solid state.[1] However, studies using the chemistry-rich heavier spin-$^1/_2$ nuclides are still quite rare. Recently several studies employing ^{113}Cd[2] NMR have appeared, and ^{77}Se,[3] ^{119}Sn,[4] ^{205}Tl,[5] and ^{129}Xe[6] NMR have also been explored.

In this communication we report results obtained by using the aforementioned solid-state NMR techniques to study ^{125}Te NMR in several solids. We report a suitable ^{125}Te CP/MAS NMR standard substance, explore ^{125}Te chemical shift tensors, and use a ^{125}Te–$^{35,37}Cl$ *J* coupling to derive structural information.

Figure 1a shows the ^{125}Te NMR spectrum[7] of the monoclinic form of telluric acid ($Te(OH)_6$) obtained with cross-polarization and dipolar decoupling. As application of magic angle spinning shows, there are two inequivalent Te atoms in the unit cell, in agreement with crystal structure data.[8] The powder pattern features can be understood in terms of two overlapping, completely general, shielding tensors: site 1, $\sigma_{11} = -47.4$, $\sigma_{22} = 11.3$, $\sigma_{33} = +36.0$, σ_{iso} $((CH_3)_2Te) = -692.2$ ppm; site 2, $\sigma_{11} = -66.0$, $\sigma_{22} = +13.4$, $\sigma_{33} = +52.6$, σ_{iso} $((CH_3)_2Te) = -685.5$ ppm. This illustrates the great sensitivity of the ^{125}Te shielding to even minor distortions from octahedral symmetry (Te–O distances lie between 1.907 and 1.914 Å; O–Te–O angles lie between 87.60° and

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[†] NRCC No. 27615.

* Author to whom correspondence should be addressed.

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(7) ^{125}Te NMR spectra were recorded at 56.8 MHz on a Bruker CXP-180 NMR spectrometer. Single cross-polarization contacts of 1–5 ms were used, with matched radio frequency field amplitudes of 45 kHz. Magic angle spinning rates of ~3 kHz were achieved with Delrin or Kel-F spinners of the Andrew–Beams type.

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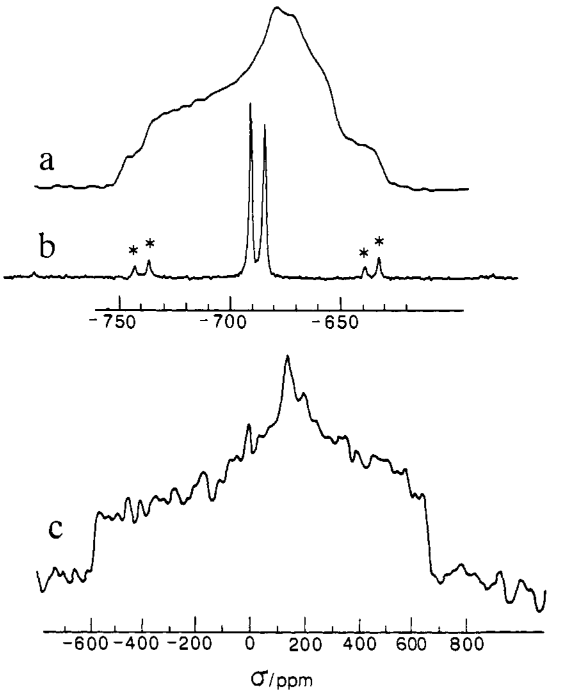

Figure 1. (a) Solid-state ^{125}Te NMR spectrum of Te(OH)$_6$, static sample, τ_{cp} = 2 ms, N_s = 1612, recycle time = 10 s. (b) Spinning sample, τ_{cp} = 2 ms, N_s = 160, recycle time = 10 s. (c) Solid-state ^{125}Te NMR spectrum of $(CH_3)_2Te$ at 77 K. Spectrum obtained with Hahn echo, N_s = 480, recycle time = 10 s.

Table I. Summary of ^{125}Te Chemical Shift (ppm) Results[a]

	σ_{11}	σ_{22}	σ_{33}	σ_{iso}	σ_{iso} (liq)
$(CH_3)_2Te$[b]	−643	62	581	69	0
Te(OH)$_6$					
site 1	−47.4	11.3	36.0	−692.2	−712[c]
site 2	−66.0	13.4	52.6	−685.5	
$(CH_3)_3TeCl\cdot H_2O$	−56.0	−1.0	57.0	−444	
$(CH_3)_3TeNO_3$	−71.5	31.5	40.0	−472	
$(CH_3)_3TeI$				−404	−443[d]

[a] Results are for solids at room temperature unless otherwise stated. [b] Solid at 77 K; the difference between σ_{iso} (solid, 77 K) and δ_{iso} (liquid, room temperature) probably is largely a temperature effect. [c] Reference 12. [d] Reference 13.

92.08°).[8] It is likely that site 2, which has the wider powder pattern, corresponds to Te(2), the site with the larger variation in Te–O distances in the crystal.

As telluric acid yields a simple spectrum in a short time, it is a suitable ^{125}Te CP/MAS standard material. It is also suitable as a secondary ^{125}Te chemical shift standard for solids, as its shift values fall approximately in the center of the usual ^{125}Te shift range.

In order to assess the order of magnitude of the anisotropic shielding to be expected for low-symmetry tellurium compounds, we examined the spectrum of dimethyl telluride at 77 K (Figure 1c). The resulting powder pattern is extremely broad (~70 KHz ≈ 1200 ppm), with σ_{11} = −643, σ_{22} = 62, and σ_{33} = 581 ppm. This suggests that there may be some advantage in performing ^{125}Te NMR on solids at lower magnetic fields.

Several other examples of solid-state ^{125}Te NMR spectra are provided by compounds containing the $(CH_3)_3Te$ moiety. Spectra for static and spinning samples of $(CH_3)_3TeNO_3$ are shown in Figure 2a and 2b. Evidently there is only one type of Te atom in the unit cell. The near-axial shielding tensor suggests that the Te atom is not on a threefold axis but that deviations from axial symmetry are small. The anisotropic shift values are summarized in Table I. Figure 2c and 2d show static and spinning ^{125}Te NMR spectra of a sample of $(CH_3)_3TeCl$ recrystallized from water. The shielding tensor is almost completely asymmetric although the moderate magnitude of anisotropic shielding suggests that deviations from regular geometry are not large. The spectrum obtained on application of magic angle spinning shows a surprising amount of fine structure. Detailed examination (Figure 2e) shows that the spectrum is a well-resolved septuplet with an average splitting of 102 Hz. This suggests that the Te atom is coupled to two equivalent spin-$^3/_2$ Cl nuclei. The intensities depart slightly from the ideal 1:2:3:4:3:2:1 ratio, as the ^{35}Cl and ^{37}Cl nuclei, with a natural abundance of 75.4% and 24.6%, respectively, have slightly different coupling constants, and Te is coupled to ^{35}Cl^{35}Cl (57%), ^{35}Cl^{37}Cl (37%), and ^{37}Cl^{37}Cl (6%) pairs. This causes the multiplet line width to increase progressively on going from the central component to the outer components. By assuming that the observed splitting is a weighted average for the two isotopes, the ^{125}Te–^{35}Cl J coupling is calculated to be 106 ± 5 Hz.

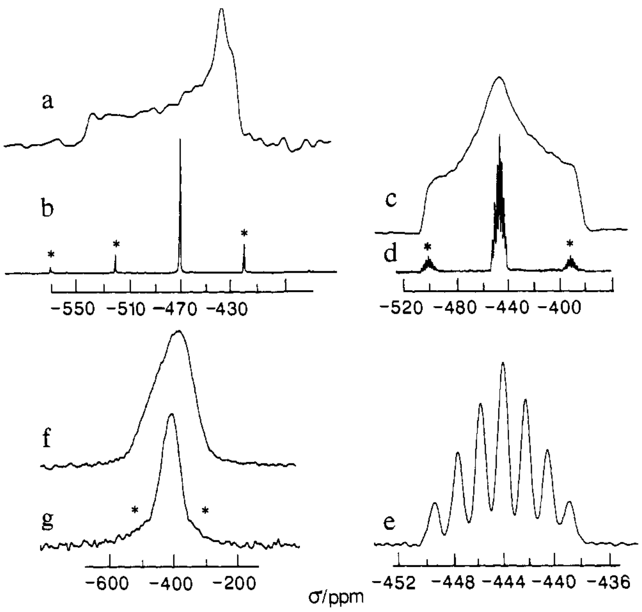

Figure 2. (a) Solid-state ^{125}Te NMR spectrum of $(CH_3)_3TeNO_3$, static sample, τ_{cp} = 2 ms, N_s = 196, recycle time = 20 s. (b) Spinning sample, τ_{cp} = 2 ms, N_s = 64, recycle time = 20 s. (c) Solid-state ^{125}Te NMR spectrum of $(CH_3)_3TeCl\cdot H_2O$, static sample, τ_{cp} = 2 ms, N_s = 1212, recycle time = 20 s. (d) Same as c, spinning sample, τ_{cp} = 2 ms, N_s = 96, recycle time = 20 s. (e) Same as d, expanded scale. (f) Solid-state ^{125}NMR spectrum of $(CH_3)_3TeI$, static sample, τ_{cp} = 5 ms, N_s = 2580, recycle time = 20 s. (g) Same as f, spinning sample, τ_{cp} = 5 ms, N_s = 176, recycle time = 20 s.

Usually the fine structure which results from coupling of a spin-$^1/_2$ nucleus to a quadrupolar nucleus is a complicated function of the dipolar coupling, J coupling, the quadrupole coupling constant, and the magnetic field strength.[9] However, recent calculations[10] have shown that for sufficiently high fields the multiplet splitting always approaches values attributable to J coupling only. We believe this ^{125}Te–^{35,37}Cl J coupling to be the first example of this phenomenon. The lack of a dipolar contribution to the splitting at only modest field strength suggests a long Te–Cl distance and/or a small Cl quadrupole coupling constant.

The ^{125}Te NMR results, together with the ^{13}C CP/MAS NMR spectrum, allow at least a partial description of the $(CH_3)_3TeCl$ structure. The ^{13}C CP/MAS spectrum shows two lines in a 2:1 ratio. This, together with the fact that the Te atom is coupled to two equivalent chlorines, suggests that two methyls and the two chlorines are related by reflection in a mirror plane which includes the Te atom and one methyl group. Diffraction studies[11]

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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_3TeI (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)

Richard D. McCullough, Gaik B. Kok, Knud A. Lerstrup, and Dwaine O. Cowan*

Department of Chemistry, The Johns Hopkins University
Baltimore, Maryland 21218

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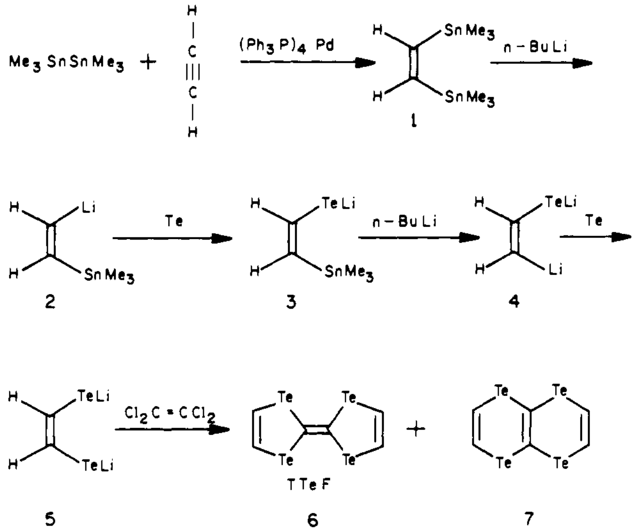
One of the long-standing goals of organic solid-state research has been the creation of new heterocyclic π-donors in an attempt to alter the electrical conductivity of radical cation salts derived from these donors.[1] Reported herein is the first synthesis of tetratellurafulvalene (TTeF, **6**). This 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, $(TMTSF)_2ClO_4$ and related salts and bis(ethylenedithio)tetrathiafulvalene (BEDT-TTF) salts, $(BEDT\text{-}TTF)_2I_3$).

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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