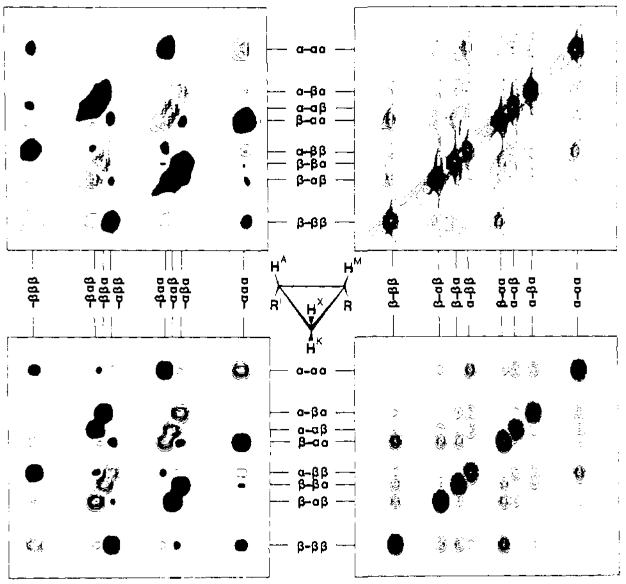

Figure 1. Multiplets taken from small flip angle NOESY spectra of *cis*-2-phenylcyclopropanecarboxylic acid ethyl ester (R = $COOCH_2CH_3$, R' = C_6H_5). The cross-peak (left) is centered at $\omega_1 = \Omega_M$ and $\omega_2 = \Omega_A$; the diagonal peak (right) is centered at $\omega_1 = \omega_2 = \Omega_M$. Top: experimental multiplets. Bottom: simulations, calculated without initial rate or weak coupling approximations but neglecting correlation effects and motional anisotropy. Experimental parameters: sequence $90°-t_1-10°-\tau_m-10°-t_2$, zero-quantum signals shifted[16] by incrementing τ_m from 3.6 to 3.7 s; spectral width 770 Hz in both domains, data matrix 1K × 4K points before, 4K × 4K points after zero filling. Parameters in simulation: $\tau_c = 6.7$ ps, hence extreme narrowing with $W_0^{ij}:W_1^{ij}:W_2^{ij} = 2:3:12$. The dipolar contributions were calculated from known proton–proton distances:[15] W_2^{AM}, W_2^{AK}, W_2^{AX}, W_2^{KM}, W_2^{KX}, W_2^{MX} = 0.015, 0.003, 0.012, 0.003, 0.069, and 0.011 s^{-1}, respectively. Uncorrelated external random field contribution $W_1^{ext} = 0.019\ s^{-1}$ for all single-quantum transitions. Equidistant contours in experimental and simulated spectra at ±3%, ±6%, ...; ±99% of the maximum peak in corresponding diagonal multiplet. Filled contours on the left represent positive signals, all other peaks being negative, including the signals on the diagonal which appear with densely spaced contours.

If $\tau_m > 0$, the populations migrate between the 2^N eigenstates of the N-spin system:

$$\Delta \boldsymbol{P}(\tau_m) = \exp\{\mathbf{W}\tau_m\}\Delta \boldsymbol{P}(0) \approx \{\mathbf{1} + \mathbf{W}\tau_m\}\Delta \boldsymbol{P}(0) \quad (1)$$

where $\Delta \boldsymbol{P}$ is the vector of the deviations of the populations from thermal equilibrium and **W** the matrix of transition probabilities. The equality holds for arbitrary duration of the mixing interval τ_m, while the approximation is restricted to the initial rate regime. Simulations of small flip angle NOESY spectra can be calculated by inserting eq 1 into our "Grandchild of Laocoon" program for simulating *z*-COSY spectra,[13] taking care that **W** must be expressed in the eigenbase.

The principle is illustrated in Figure 1, which shows multiplets taken from small flip angle NOESY spectra of a disubstituted cyclopropane derivative. The transitions are labeled by shorthand expressions for the corresponding products[5,14] of shift operators I^+, I^- and polarization operators I^α, I^β. Thus, $\{\alpha-\beta\alpha\}$ stands for $I_A^\alpha I_M^- I_K^\beta I_X^\alpha$ (the four spins always appear in the same order.) In the *diagonal multiplet* of spin M in Figure 1, the predominant off-diagonal peaks are due to the interactions of the geminal protons K and X (r_{KX} = 1.79 Å),[15] notably a strong peak connecting $\{\alpha-\alpha\alpha\}$ and $\{\alpha-\beta\beta\}$, proportional to W_2^{KX} in the initial rate approximation, since protons K and X must flip simultaneously. The peak connecting $\{\alpha-\alpha\alpha\}$ and $\{\beta-\alpha\beta\}$ is much weaker, reflecting a greater separation (r_{AX} = 2.44 Å), while there is no detectable peak connecting $\{\alpha-\alpha\alpha\}$ and $\{\beta-\beta\alpha\}$ (r_{AK} = 3.0 Å). Other peaks carry information about single-quantum transition probabilities, such as the peak connecting $\{\alpha-\alpha\alpha\}$ and $\{\alpha-\beta\alpha\}$ (proportional to W_{1K}). The *cross-peak multiplet* connecting A and M (left half of Figure 1) features strong signals between connected transitions, e.g., between $\{\alpha-\alpha\alpha\}$ and $\{-\alpha\alpha\alpha\}$, which occur in the absence of relaxation, like in *z*-COSY.[12,13] The peak between $\{\alpha-\alpha\alpha\}$ and $\{-\beta\beta\beta\}$ is proportional to W_2^{KX} (in the initial rate approximation), since, in addition to the permutation of A and M in the roles of active and passive spins, this peak reflects concerted flips of K and X. Other peaks have amplitudes that reflect the superposition of various pathways. For example, the amplitude of the signal correlating the transitions $\{\beta-\beta\alpha\}$ and $\{-\alpha\alpha\alpha\}$ can be rationalized as follows: since $\{\beta-\beta\alpha\}$ is converted into populations $\{\beta\alpha\beta\alpha\}$ and $\{\beta\beta\beta\alpha\}$, while the coherence $\{-\alpha\alpha\alpha\}$ is proportional to the difference of the populations $\{\alpha\alpha\alpha\alpha\}$ and $\{\beta\alpha\alpha\alpha\}$, there are three relaxation pathways that may contribute to the signal: W_2^{AK} connects $\{\beta\alpha\beta\alpha\}$ and $\{\alpha\alpha\alpha\alpha\}$, W_2^{MK} leads from $\{\beta\beta\beta\alpha\}$ to $\{\beta\alpha\alpha\alpha\}$, while W_1^K connects $\{\beta\alpha\beta\alpha\}$ to $\{\beta\alpha\alpha\alpha\}$. As a result, the cross-peak corresponding to the process $\{\beta-\beta\alpha\} \rightarrow \{-\alpha\alpha\alpha\}$ is proportional to $W_2^{AK} + W_2^{MK} - W_1^K$.

A quantitative evaluation of the **W** matrix requires a comparison of experimental and simulated multiplets. The dipolar contributions to the **W** matrix were calculated from known proton–proton distances.[15] Only the isotropic correlation time, τ_c, and the amount of external (uncorrelated) random field relaxation were adjusted empirically to obtain satisfying agreement between theory and experiment. No agreement could be found if the assignments of the K and X resonances were inverted. Thus, the small flip angle NOESY method allows one to solve stereochemical questions such as the distinction between cis and trans isomers in our example. With this experiment one obtains information about both scalar and dipolar couplings (COSY and NOESY information) in one single spectrum.

Acknowledgment. We are indebted to R. R. Ernst, in whose laboratory preliminary experiments were carried out, and to Peter Pfändler for assistance in modifying the Grandchild of Laocoon program. This research was supported in part by the Swiss National Science Foundation (Grant 2.925-0.85.)

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Chemistry of Cationic Zirconium(IV) Benzyl Complexes. One-Electron Oxidation of d^0 Organometallics

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Cationic d^0 complexes $Cp_2M(R)^+$ ($Cp^- = C_5H_5^-$, M = Ti, Zr) have been implicated as active species in soluble Cp_2MCl_2/AlR_nCl_{3-n} Ziegler–Natta olefin polymerization catalyst systems.[1,2] We recently showed that the THF complex $[Cp_2Zr(CH_3)(THF)][BPh_4]$ (**1**) polymerizes ethylene in CH_2Cl_2 solvent under mild conditions.[2] The active species in this reaction is probably the "naked alkyl" $Cp_2Zr(R)^+$ since THF inhibits the polymerization, and **1** does not coordinate a second THF ligand.[3] We now report that the benzyl analogue of **1**, $[Cp_2Zr(CH_2Ph)(THF)][BPh_4]$ (**2**), can be prepared by a novel one-electron oxidation of d^0 $Cp_2Zr(CH_2Ph)_2$, and that **2** also polymerizes ethylene.

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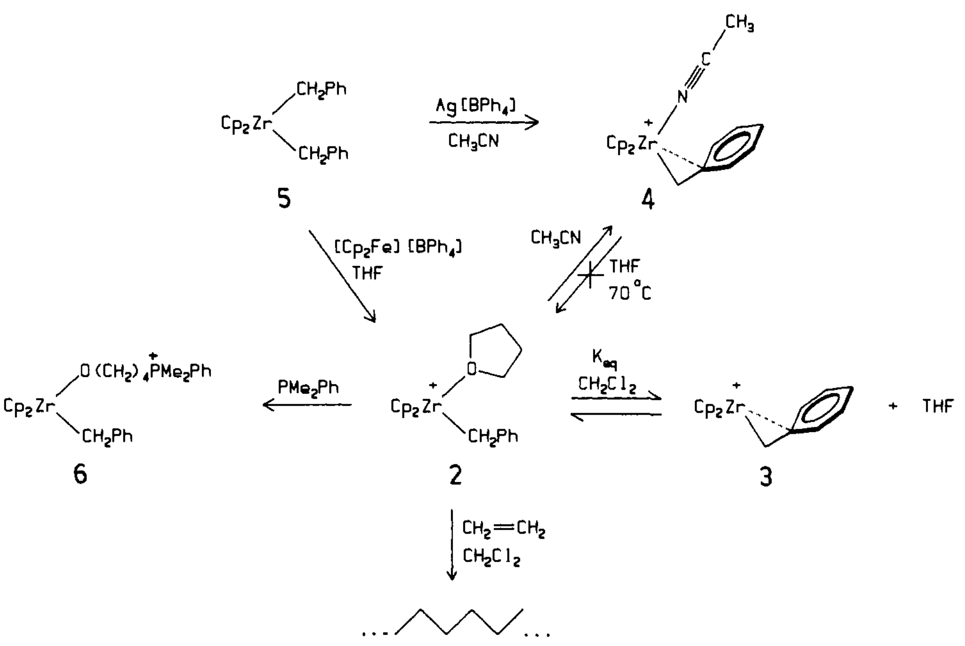
Scheme I



Complex **2** undergoes significant THF dissociation in CH_2Cl_2 to produce the naked benzyl complex $[Cp_2Zr(\eta^2\text{-}CH_2Ph)][BPh_4]$ (**3**) (or its CH_2Cl_2 solvate) which is stabilized by multidentate Zr–benzyl bonding and which can be directly observed by ^1H NMR spectroscopy.[4]

The cationic benzyl complex $[Cp_2Zr(\eta^2\text{-}CH_2Ph)(CH_3CN)][BPh_4]$ (**4**) was prepared as a yellow crystalline solid by the reaction of $Cp_2Zr(CH_2Ph)_2$ (**5**)[5] with $Ag[BPh_4]$ in CH_3CN (eq 1).[3,6] Details of the Zr–benzyl bonding in **4** were established by

$$\underset{\mathbf{5}}{Cp_2Zr(CH_2Ph)_2} + Ag[BPh_4] \xrightarrow{CH_3CN} \underset{\mathbf{4}}{[Cp_2Zr(\eta^2\text{-}CH_2Ph)(CH_3CN)][BPh_4]} + Ag^0 + {}^1\!/_2PhCH_2CH_2Ph \quad (1)$$

a single-crystal X-ray diffraction study.[7] The $Cp_2Zr(\eta^2\text{-}CH_2Ph)(CH_3CN)^+$ cation adopts the normal bent metallocene structure with the CH_3CN and $PhCH_2^-$ ligands arrayed in the plane between the two Cp^- ligands as shown in Figure 1. The benzyl ligand is bonded to Zr in an η^2 fashion thru both the methylene carbon and the phenyl π system, as evidenced by the acute Zr–C(0)–C(1) angle and the short Zr–C(1) distance. Unusually high-field ^1H NMR resonances for the ortho hydrogens (δ 6.7)[8] and ^{13}C NMR resonances for the methylene and ipso carbons (δ 44.1, 126.0 vs. 60.7, 153.0 for **5**) indicate that this structure is maintained in solution.[9] Similar structures have been observed for other d^0 and $d^0 f^n$ metal benzyl complexes.[10]

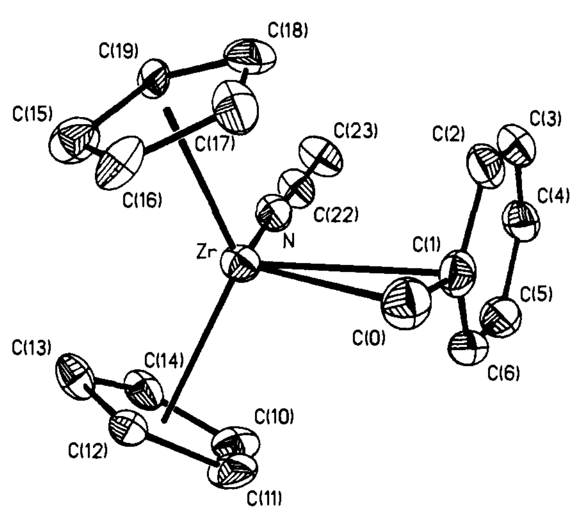


Figure 1. Structure of the $Cp_2Zr(\eta^2\text{-}CH_2Ph)(CH_3CN)^+$ cation ($\mathbf{4}^+$). Important interatomic distances (angstroms) and bond angles (deg) are as follows: Zr–C(0), 2.344 (8); Zr–C(1), 2.648 (6); Zr–C(2), 3.252 (8); Zr–C(6), 3.236 (8); Zr–N, 2.295 (5); N–C(22), 1.122 (7); C(22)–C(23), 1.438 (8); Zr–CNT(1), 2.190; Zr–CNT(2), 2.167; Zr–C(0)–C(1), 84.9 (4); N–Zr–C(0), 115.1 (2); C(0)–Zr–C(1), 33.3 (2); C(22)–N–Zr, 173.5 (4); CNT(1)–Zr–CNT(2), 128.4 (CNT denotes the centroid of a Cp ring).

Complex **4** does not react with ethylene. We reasoned that replacement of the coordinated CH_3CN with a weaker ligand should promote ligand dissociation and ethylene polymerization and allow observation or isolation of the naked benzyl species $[Cp_2Zr(\eta^2\text{-}CH_2Ph)][BPh_4]$ (**3**). Initial attempts to prepare other

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[4] $Cp_2Ti(CH_3)(L)^+$ complexes,[a] neutral $(Cp')_2MR$ (M = group 3, lanthanide) complexes,[b–g] and other cationic, d^0 complexes[h–k] have been investigated: (a) Bochmann, M.; Wilson, L. M. *J. Chem. Soc., Chem. Commun.* **1986**, 1610. (b) Watson, P. L.; Parshall, G. W. *Acc. Chem. Res.* **1985**, *18*, 51. (c) Bercaw, J. E.; Davies, D.; Wolczanski, P. T. *Organometallics* **1986**, *5*, 443. (d) Thompson, M. E.; Baxter, S. M.; Bulls, A. R.; Burger, B. J.; Nolan, M. C.; Santarsiero, B. D.; Schaefer, W. P.; Bercaw, J. E. *J. Am. Chem. Soc.* **1987**, *109*, 203. (e) Jeske, G.; Lauke, H.; Mauermenn, H.; Swepston, P. N.; Schumann, H.; Marks, T. J. *J. Am. Chem. Soc.* **1985**, *107*, 8091. (f) Evans, W. J.; Meadows, J. H.; Hunter, W. E.; Atwood, J. L. *J. Am. Chem. Soc.* **1984**, *106*, 1291. (g) Evans, W. J.; Dominguez, R.; Hanusa, T. P. *Organometallics* **1986**, *5*, 263. (h) Wengrovius, J. H.; Schrock, R. R. *Organometallics* **1982**, *1*, 148. (i) Kress, J.; Osborn, J. A. *J. Am. Chem. Soc.* **1983**, *105*, 6346. (j) Schrock, R. R.; Sharp, P. R. *J. Am. Chem. Soc.* **1978**, *100*, 2389. See also: (k) Toscano, P. J.; Marks, T. J. *J. Am. Chem. Soc.* **1985**, *107*, 653.

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[7] Crystallographic data: a = 10.252 (3) Å, b = 11.681 (3) Å, c = 14.788 (4) Å, β = 103.41 (2)°, V = 1722.7 (8) Å^3, Z = 2 in space group $P2_1$; R = 0.040, R_w = 0.053 for 2718 unique reflections with $I \geq 3\sigma(I)$. The Cp^- rings and the BPh_4^- phenyl groups were treated as rigid bodies.

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[9] Data for **2–6** are given as supplementary material.

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$Cp_2Zr(CH_2Ph)(L)^+$ 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)

$$\underset{\mathbf{5}}{Cp_2Zr(CH_2Ph)_2} + [Cp_2Fe][BPh_4] \xrightarrow{THF} \underset{\mathbf{2}}{[Cp_2Zr(CH_2Ph)(THF)][BPh_4]} + Cp_2Fe + {}^1/_2PhCH_2CH_2Ph \quad (2)$$

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.

Acknowledgment. Support for this work was provided by the Research Corporation, the donors of the Petroleum Research Fund, administered by the American Chemical Society, and Washington State University. The X-ray diffraction system was acquired with the aid of NSF Grant CHE8408407 and the Boeing Co. Evan Lewis performed some early experiments.

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[†]

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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.

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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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