



Synthesis and characterization of dicyclohexyl telluride; Raman spectra and multinuclear NMR studies of dialkyl tellurides

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Abstract

Dicyclohexyl telluride, $\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$, a somehow surprisingly not reported molecule, is synthesized and characterized by multinuclear NMR, IR and Raman spectroscopy. A temperature dependence of the NMR spectra ($^{125}\text{Te}\{^1\text{H}\}$, $^{13}\text{C}\{^1\text{H}\}$) for $\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$ is observed and studied in detail. The ^{125}Te -NMR spectrum of $\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$ at different temperatures is calculated and compared with the experimental spectra. The vibrational spectra of $\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$ and values calculated ab initio at HF/3-21G* level are discussed. The Raman spectra of symmetric dialkyl tellurides TeR_2 with $\text{R}_2 = (n\text{-C}_3\text{H}_7)_2$, $(i\text{-C}_3\text{H}_7)_2$, $(n\text{-C}_4\text{H}_9)_2$ and $(n\text{-C}_6\text{H}_{13})_2$ are reported and TeC stretching frequencies assigned. In addition, thorough NMR characterization of those derivatives ($^{125}\text{Te}\{^1\text{H}\}$, $^{13}\text{C}\{^1\text{H}\}$, ^1H) is given. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Dialkyl tellurides; Raman spectroscopy; Semiempirical calculations; ^{125}Te -NMR; Variable temperature NMR calculation

1. Introduction

Although the first diorgano tellurium compound, diethyl telluride, was synthesized in 1840 [1], organotellurium chemistry has developed slowly. Studies and research were complicated by the low stability of many compounds, which were often light and air sensitive and frequently of hateful odor [2]. Currently, organotellurium compounds have attracted a great interest as useful reagents in organic synthesis and nuclear medicine [3–7] as well as precursors for the organometallic vapor-phase epitaxial (OMVPE) film growth of semiconductor materials [8,9].

Many dialkyl tellurides are known so far, but structural characterization by common spectroscopic methods is rather limited [10]. However, to our knowledge, dicycloalkyl tellurides are not known. The only reports

about dicyclohexyl tellurium compounds deal with dicyclohexyl substituted telluronium salts [3] (noting very briefly dicyclohexyl telluride as a precursor) or more recently with the stepwise formation of $\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$ as a ligand in an osmium cluster [11] or with tellurium(IV) compounds [12]. This prompted us to synthesize and characterize dicyclohexyl telluride as a representative member of dicycloalkyl tellurides.

Characterization by vibrational spectroscopy of dialkyl tellurides is limited. Up to now, Raman spectra are reported only for $\text{Te}(\text{CH}_3)_2$, $\text{Te}(\text{C}_2\text{H}_5)_2$ and $\text{Te}(t\text{-C}_4\text{H}_9)_2$ [13,14] and detailed IR data only for $\text{Te}(\text{CH}_3)_2$ [15]. Here we report the Raman spectra of some higher dialkyl tellurides. For the new dicyclohexyl telluride we discuss the IR/Raman spectra together with values calculated ab initio at HF/3-21G* level [16]. Detailed NMR data of dialkyl tellurides TeR_2 ($\text{R}_2 = (n\text{-C}_3\text{H}_7)_2$; $(i\text{-C}_3\text{H}_7)_2$; $(n\text{-C}_4\text{H}_9)_2$; $(n\text{-C}_6\text{H}_{13})_2$) are given. Variable temperature NMR studies of $\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$ reveal an interesting behavior.

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2. Results and discussion

The reaction of disodium telluride, Na_2Te , with alkyl halides is the oldest known method for the synthesis of dialkyl tellurides [17,18]. Na_2Te can be prepared easily from stoichiometric amounts of tellurium and sodium in tetrahydrofuran in the presence of naphthalene in nearly quantitative yields. Reported reaction times vary from some hours to 4 days [15,19–22]. Initially, the mixture turns dark green because of the formation of sodium naphthalide [19] and then becomes pale brown. The alkyl halide can be added either to a solution of isolated Na_2Te or to in situ generated Na_2Te . It appears, that the reaction time for the formation of Na_2Te depends on the size (surface) of the used sodium and tellurium ingots. Short reaction times require small pieces of sodium and tellurium powder. Sodium telluride is a pale brown and highly air sensitive powder, insoluble in hexane and tetrahydrofuran but soluble in alcohols. A solution with methanol or ethanol has a purple color because it is assumed that traces of oxygen present oxidize Na_2Te to Na_2Te_2 [19].

We achieved nearly quantitative formation of Na_2Te by reacting sodium pieces with tellurium powder in THF with catalytic amounts of naphthalene for 2 days at 25°C. Successful synthesis of Na_2Te in almost quantitative yields, as reported for in situ preparation of dialkyl tellurides, could not be reproduced [20].

2.1. Vibrational spectroscopy and calculations

The Raman frequencies of νTeC for TeR_2 ($\text{R}_2 = (n\text{-C}_3\text{H}_7)_2$; $(i\text{-C}_3\text{H}_7)_2$; $(n\text{-C}_4\text{H}_9)_2$; $(n\text{-C}_6\text{H}_{13})_2$; $(\text{cyclo-C}_6\text{H}_{11})_2$) as well as for the reported $\text{Te}(\text{CH}_3)_2$, $\text{Te}(\text{C}_2\text{H}_5)_2$ and $\text{Te}(t\text{-C}_4\text{H}_9)_2$ are given in Table 1. The complete spectra are listed in Section 3. The characteristic TeC stretching frequencies (ν_s) of TeR_2 are found in the Raman spectra in the region around 500 cm^{-1} and are of medium to strong intensity. As expected, the highest frequency for ν_s is observed for $\text{Te}(\text{CH}_3)_2$ (522/

Table 1
TeC stretching frequencies (cm^{-1}) (Raman) of dialkyl tellurides

TeR_2 (l)		$\nu_{\text{as}} \text{ TeC}$	$\nu_s \text{ TeC}$
$\text{Te}(\text{CH}_3)_2$	[13]	–	523 ^a
	[14]	–	522 ^b
$\text{Te}(\text{C}_2\text{H}_5)_2$		516	502
	[14]	–	500 ^b
$\text{Te}(n\text{-C}_3\text{H}_7)_2$		596	505
$\text{Te}(i\text{-C}_3\text{H}_7)_2$		487	509
$\text{Te}(n\text{-C}_4\text{H}_9)_2$		599	506
$\text{Te}(t\text{-C}_4\text{H}_9)_2$	[14]	–	501 ^b
$\text{Te}(n\text{-C}_6\text{H}_{13})_2$		603	507
$\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$		665	496

^a Gas phase: 535(ν_{as} , vs)/525(ν_s , sh).

^b ν_{as} and ν_s not resolved.

Table 2

Calculated and observed IR and Raman spectra of dicyclohexyl telluride

HF ν (IR/Raman) (cm^{-1}) ([rel. int.])	HF $\nu * 0.91$ (cm^{-1})	ν (IR, l) (cm^{-1})	ν (Raman, l) (cm^{-1})
3232 (23/157)	2941		
3231 (129/68)	2940	2925 (vs)	2932 (100)
3207 (42/112)	2918		2932 (100)
3195 (24/142)	2908		
3178 (26/182)	2892		2884 (46)
3177 (34/127)	2891		2850 (85)
1649 (0/60)	1501	1446 (vs)	
1646 (9/19)	1498		1440 (26)
1509 (24/2)	1373	1342 (m)	1333 (17)
1424 (2/50)	1296		1299 (10)
1421 (50/2)	1293	1292 (w)	
1417 (0/23)	1290		1257 (22)
1333 (4/43)	1213	1167 (s)	1173 (31)
1320 (19/14)	1201	1246 (s)	1173 (31)
1210 (6/1)	1101	1080 (m)	1084 (11)
1124 (10/4)	1023	1024 (w)	1025 (24)
1092 (9/9)	993	990 (s)	991 (16)
944 (16/3)	859	861 (w)	846 (25)
909 (3/7)	827	804 (w)	807 (31)
706 (1/10)	643	661 (m)	665 (50)
687 (8/3)	626	636 (w)	640 (13)
548 (1/33)	499	493 (w)	496 (26)
497 (1/12)	452		454 (21)
			224 (51)
240 (0/6)	218		201 (41)

523 cm^{-1}). A second absorption of nearly equal intensity at ca. 90 cm^{-1} higher frequency, observed for the higher di-*n*-alkyl tellurides, is tentatively assigned to the asymmetric stretching frequencies (ν_{as}). The respective literature values of ν_{as} for $\text{Te}(\text{CH}_3)_2$, $\text{Te}(\text{C}_2\text{H}_5)_2$ and $\text{Te}(t\text{-C}_4\text{H}_9)_2$ are too close to be resolved [13,14] (for a detailed discussion see Ref. [14]). For $\text{Te}(\text{C}_2\text{H}_5)_2$ we found a very strong absorption at 502 cm^{-1} (ν_s) with a shoulder at 516 cm^{-1} which we assign to the asymmetric TeC stretching frequency. It appears that for longer chain di-*n*-alkyl tellurides the energy difference between ν_{as} and ν_s increases significantly. For $\text{Te}(i\text{-C}_3\text{H}_7)_2$ and $\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$ absorptions at 487 and 665 cm^{-1} , respectively, clearly different from those of the *n*-alkyl derivatives due to the changed environment of the center tellurium, are found and assigned to ν_{as} . Definite assignments of the TeC_2 deformation mode in the region of 200 cm^{-1} , as observed for $\text{Te}(\text{CH}_3)_2$ at 194 [13]/195 [14] cm^{-1} , cannot be made.

For dicyclohexyl telluride all observed and calculated Raman and IR frequencies are given in Table 2. The symmetric TeC stretching mode occurring in the Raman spectrum at 496 cm^{-1} is observed in the IR spectrum as a weak absorption at 493 cm^{-1} .

The structure, energies and vibrational frequencies of $\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$ were calculated semiempirically at PM3/VSTO-3G* level and ab initio (all-electron calcu-

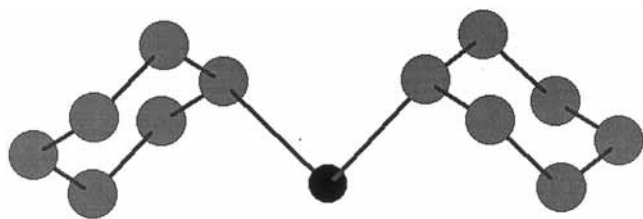


Fig. 1. Structure of $\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$ computed ab initio at HF/3-21G* level of theory, optimized fully within C_1 symmetry.

lation) at HF/3-21G* level of theory. The calculated structure represents at both levels a true minimum (NIMAG = 0, for ab initio calculated frequencies see Table 2). At HF/3-21G* level the structure of $\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$ was optimized fully within C_1 symmetry (Fig. 1). The total energy was computed to be $E^{\text{HF}} = -7047.931082$ a.u. with the zero point energy (*zpe*) being $215.9 \text{ kcal mol}^{-1}$. The two Te–C bond distances were calculated to be 2.200 \AA with a C–Te–C bond angle of 94.3° .

The values of the calculated vibrational frequencies have to be scaled with a factor 0.91 [16,23]. The transformation results for ν_s in a calculated value of 499 cm^{-1} which agrees well with the experimental data. For the tentative assignment of ν_{as} at 665 cm^{-1} in the Raman spectrum and 661 cm^{-1} in the IR spectrum a calculated value of 643 cm^{-1} shows only moderate agreement. All in all there is good correlation between calculated and observed data.

2.2. NMR spectroscopy

A thorough NMR characterization of the dialkyl tellurides described in our work is given in Table 3 and

includes the data for the lower members $\text{Te}(\text{CH}_3)_2$ and $\text{Te}(\text{C}_2\text{H}_5)_2$. Some of the data have not been reported yet. The most convenient nucleus for the characterization of these materials is ^{125}Te . The tellurium chemical shifts in TeR_2 depend highly on the nature of R. With increasing number of methylene groups present in di-*n*-alkyl tellurides, their influence on $\delta^{125}\text{Te}$ diminishes, as observed in the series (Me 0, Et 374, *n*-Pr 233, *n*-Bu 249, *n*-Hex 249 ppm). For R = isopropyl (696 ppm) and cyclohexyl (592 ppm), respectively, a larger downfield shift compared to *n*-propyl and *n*-hexyl is observed. This is in agreement with the observed chemical shifts of various dialkyl tellurides discussed by O'Brien et al. where also, based on their results, predictions of chemical shifts for ^{125}Te were made [22].

In the ^{13}C -NMR spectra the carbon atoms bonded to tellurium show the typical high field resonance and ^{125}Te satellites with $^1J_{\text{C-Te}}$ coupling constants of 145–155 Hz.

2.3. NMR studies of dicyclohexyl telluride

An interesting NMR phenomenon is observed in the ^{125}Te - and ^{13}C -NMR spectra of dicyclohexyl telluride. The ring inversion of the cyclohexyl rings causes a temperature dependency of the observed resonances.

The ^{125}Te -NMR spectrum of dicyclohexyl telluride shows at room temperature an unusual broad resonance at $\delta = 592 \text{ ppm}$. Variable temperature ^{125}Te -NMR spectra in CDCl_3 as well as in toluene- d_8 were recorded. The spectra obtained from toluene- d_8 solutions are displayed in Fig. 2. The ^{125}Te resonance of $\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$ at ambient temperature is visible only in concentrated solutions requiring still a high number of scans.

Table 3
NMR data of TeR_2 in CDCl_3

δ (ppm), J (Hz)	$\text{Te}(\text{CH}_3)_2$	$\text{Te}(\text{C}_2\text{H}_5)_2$	$\text{Te}(\textit{n}\text{-C}_3\text{H}_7)_2$	$\text{Te}(\textit{i}\text{-C}_3\text{H}_7)_2$	$\text{Te}(\textit{n}\text{-C}_4\text{H}_9)_2$	$\text{Te}(\textit{n}\text{-C}_6\text{H}_{13})_2$	$\text{Te}(\textit{c}\text{-C}_6\text{H}_{11})_2$
^1H							
CH_2 (m)		2.62	2.58/1.72		2.61/1.70/1.36	2.60/1.73/1.29	2.06/1.64/1.32
CH				3.38 (sept)			3.25 (m)
CH_3	1.86 (s)	1.59 (t)	0.94 (t)	1.61 (d)	0.90 (t)	0.86 (m)	
$^3J_{\text{H-H}}$	20.2 ($^2J_{\text{H-Te}}$)	7.6	7.3	6.8	7.3		
$^{13}\text{C}\{^1\text{H}\}$							
C–Te	–21.6	–6.1	5.1	10.7	2.3	2.7	22.4
$^1J_{\text{C-Te}}$	155.2	147.9	151.0	145.3	150.5	150.5	153.6
C-2			25.5		25.1	22.5	37.1
$^2J_{\text{C-Te}}$			11.4		11.4	10.9	19.7
C-3					34.4		28.0
$^3J_{\text{C-Te}}$					10.4		–
C-3/C-4/C-5						32.2, 31.7, 31.2	25.8
$^nJ_{\text{C-Te}}$						10.4, 10.9, 10.9	31.7
CH_3		17.6	16.6	27.4	13.4	14.0	
$^nJ_{\text{C-Te}}$		11.9	11.9	26.0	–	–	
$^{125}\text{Te}\{^1\text{H}\}$	0	374	233	696	249	249	592 (br)

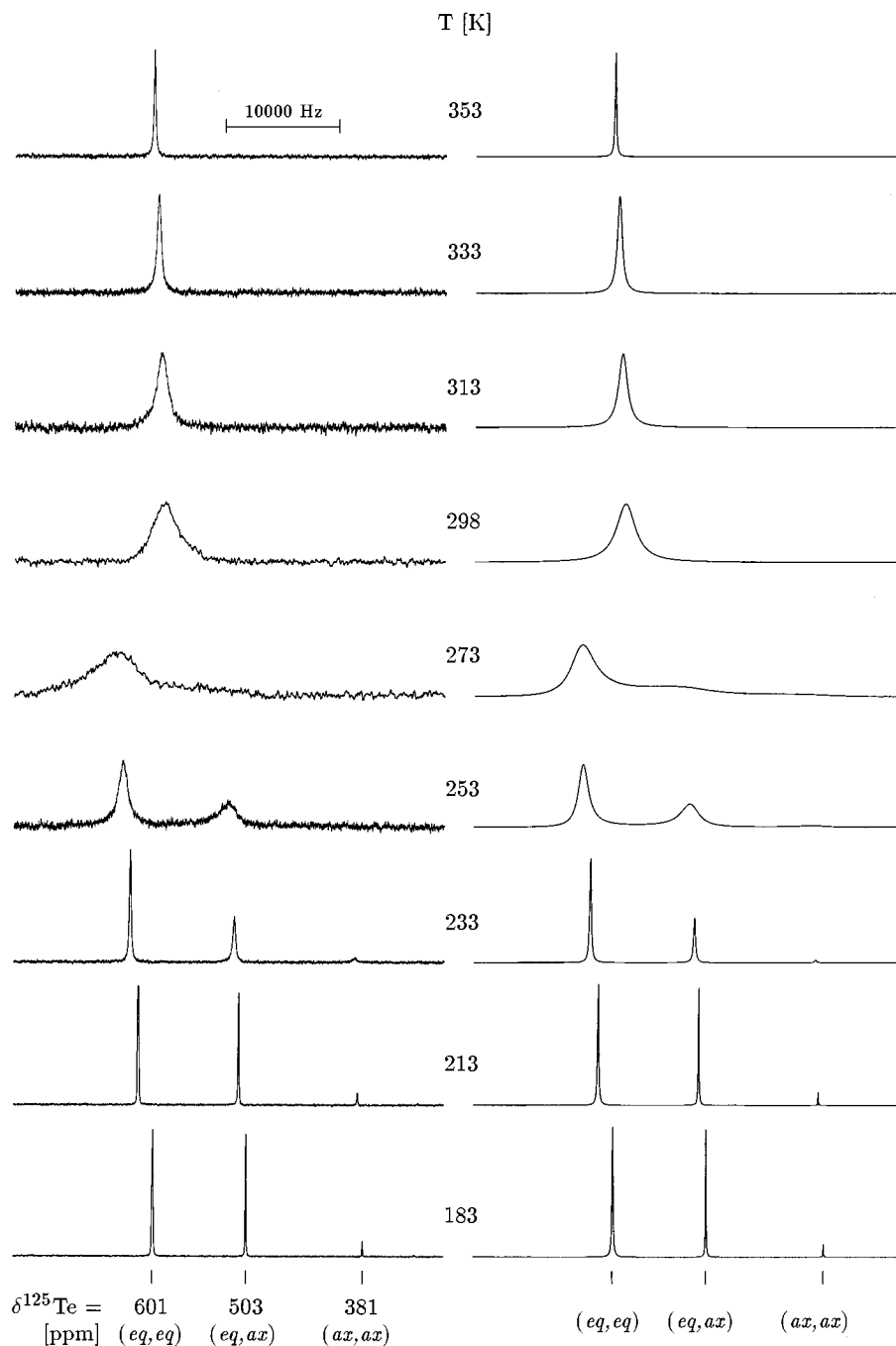
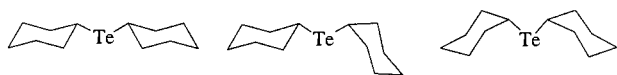


Fig. 2. Experimental (left) and calculated (right) $^{125}\text{Te}\{^1\text{H}\}$ -NMR spectra of $\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$ at variable temperature. Experimental spectra in toluene- d_8 (85.24 MHz, ca. 1 M, 30° pulse width, 0.8 s repetition rate, typically ~ 1500 scans except spectra at 253, 273 and 298 K, for which in order to obtain a satisfactory signal noise ratio ~ 3500 scans were necessary). For the calculation of the spectra the DNMR5 [27] program was used.

At ambient temperature (298 K) the ^{125}Te -NMR spectrum shows a broad signal ($\Delta\nu_{1/2} \approx 2000$ Hz) centered at $\delta = \sim 590$ ppm. It sharpens on increasing the temperature and at 353 K its half height width is only 100 Hz. Decreasing the temperature results in splitting into three signals, which again become sharp as the temperature is further lowered (Fig. 2). An analogous temperature dependence is reported for the ^{77}Se -NMR

spectra of $\text{PhSe}(\text{cyclo-C}_6\text{H}_{11})$ [24] and $\text{Se}_2(\text{cyclo-C}_6\text{H}_{11})_2$ [25] as well as for the ^{125}Te -NMR spectrum of $\text{PhTe}(\text{cyclo-C}_6\text{H}_{11})$ [26], and can be found also in the ^{125}Te -NMR spectrum of $\text{Te}_2(\text{cyclo-C}_6\text{H}_{11})_2$ [27]. In all these cases the observed temperature dependence is caused by the inversion of the cyclohexyl rings, which slows down on the NMR time scale at low temperatures. For $\text{Te}(\text{cyclo-C}_6\text{H}_{11})_2$ three conformers are possible, de-

pending on the position adopted by the tellurium atom with respect to the cyclohexyl rings: equatorial (*eq,eq*-Te(*cyclo*-C₆H₁₁)₂) or axial (*ax,ax*-Te(*cyclo*-C₆H₁₁)₂) with respect to both rings or equatorial with respect to one and axial with respect to the other ring (*eq,ax*-Te(*cyclo*-C₆H₁₁)₂).



eq,eq-Te(*cyclo*-C₆H₁₁)₂ *eq,ax*-Te(*cyclo*-C₆H₁₁)₂ *ax,ax*-Te(*cyclo*-C₆H₁₁)₂

The three signals, observed in the ¹²⁵Te-NMR spectrum at low temperatures, can be assigned to these three conformers. At 183 K the ratio *eq,eq*-Te(*cyclo*-C₆H₁₁)₂ ($\delta = 601$ ppm): *eq,ax*-Te(*cyclo*-C₆H₁₁)₂ ($\delta = 503$ ppm): *ax,ax*-Te(*cyclo*-C₆H₁₁)₂ ($\delta = 381$ ppm) estimated from the integrals is 25:14:1. The assignment of the ¹²⁵Te chemical shifts is based on the diamagnetic γ -*gauche* effect of the CH₂ groups [24,26], which is 49 ppm for the *eq,ax*-conformer and with 61 ppm even larger for the *ax,ax*-conformer. The observed values are in the same order of magnitude as for PhTe(*cyclo*-C₆H₁₁) (65 ppm [26]) and larger as for PhSe(*cyclo*-C₆H₁₁) (35–40 ppm [24]).

The ¹²⁵Te-NMR signals of the three conformers of Te(*cyclo*-C₆H₁₁)₂ display a pronounced shift to lower field with increasing temperature. From the spectra between 183 and 233 K low field shifts of 373 Hz/10 K for *eq,eq*-Te(*cyclo*-C₆H₁₁)₂, 210 Hz/10 K for *eq,ax*-Te(*cyclo*-C₆H₁₁)₂ and 127 Hz/10 K for *ax,ax*-Te(*cyclo*-C₆H₁₁)₂ can be determined. Thus the temperature dependence of $\delta^{125}\text{Te}$ is largest for the *eq,eq*-conformer and much less pronounced for the *ax,ax*-conformer. It is in the same order of magnitude found for tellurides TeR₂ (R = alkyl, aryl) [27].

The observed ¹²⁵Te-NMR spectra between 183 and 353 K could be fitted and satisfactory reproduced using the DNMR5 program [28] on the basis of an equilibrium between the three conformers (Fig. 2). For temperatures above 233 K the chemical shifts of the conformers were extrapolated assuming a linear dependency on *T* and kept constant in iteration. The activation parameters for the inversion of the cyclohexyl ring in the equilibrium between *eq,eq*-Te(*cyclo*-C₆H₁₁)₂ and *eq,ax*-Te(*cyclo*-C₆H₁₁)₂ resulting from an Eyring plot [29] are $\Delta H^\ddagger = 11.7 \pm 0.3$ kcal mol⁻¹ and $\Delta S^\ddagger = 3.6 \pm 1$ cal mol⁻¹·K⁻¹. These values fit well to those estimated for the corresponding ring inversion in PhSe(*cyclo*-C₆H₁₁) [24] and PhTe(*cyclo*-C₆H₁₁) [26] and support the well known fact, that the inversion of the cyclohexyl ring is virtually independent of the nature of the attached substituent [29].

The same effects are visible in the ¹³C-NMR spectra at variable temperatures. The spectra in toluene-*d*₈ at three significant temperatures are shown in Fig. 3. At

room temperature the resonances of the tellurium bonded carbon (C-1, 21.9 ppm) as well as the C-3/5 carbon atoms at 28.0 ppm, are slightly broadened relative to the resonances of the C-2/6 carbon atoms (37.3 ppm) and the C-4 carbon atom (26.0 ppm). Increasing the temperature beyond 298 K results in the sharpening of all four ¹³C resonances. Upon cooling, line-broadening increases and at 213 K several new resonances appear (Fig. 3). The resonances of C-1 are shifted to higher field and partly overlap with the septet of the CD₃ resonance of the solvent. At 183 K sharp resonances of the three possible conformers (*eq,eq*-; *eq,ax*-; *ax,ax*-) are visible. In the case of C-2/6 we assign the intense resonance at lower field at 37.5 ppm to the *eq,eq*-conformer and the weak resonance at the higher field at 35.4 ppm to the *ax,ax*-conformer. The two resonances for the *eq,ax*-conformer for C-2/6 are found with equal intensity at 37.2 and 36.1 ppm. These assignments are in agreement with the results obtained from the ¹²⁵Te-NMR experiments.

3. Experimental

Ethanol was freshly distilled from sodium and benzophenone. All tellurides were prepared under argon atmosphere and stored in the dark under argon at 0°C. Tellurium powder and alkyl bromides were used as commercially available from Aldrich Chemical.

The ¹²⁵Te{¹H}-, ¹³C{¹H}- and ¹H-NMR spectra were obtained as CDCl₃ solutions (plus toluene-*d*₈ for Te(*cyclo*-C₆H₁₁)₂) on Jeol GSX 270 and Jeol EX 400 instruments. Chemical shifts are reported with respect to (CH₃)₄Si (¹H, ¹³C) and (CH₃)₂Te (¹²⁵Te). The infrared spectrum was recorded as neat liquid between KBr plates on a Nicolet 520 FT-IR spectrometer. Raman spectra were recorded as neat liquids on a Perkin-Elmer Spectrum 2000 NIR FT-Raman spectrometer equipped with a Nd:YAG laser (1064 nm). The elemental analysis was performed with a C, H, N-Analysator Elementar Vario EL. The mass spectrum was obtained with a Finnigan MAT 95Q spectrometer.

The structure of Te(*cyclo*-C₆H₁₁)₂ was pre-optimized using semiempirical calculations. The calculation was carried out with the program package HyperChem [30] at the semiempirical PM3 [31] level of theory using a VSTO-3G* basis set. The PM3 method (which differs from AM1 [32] only in the values of the parameters) was chosen since the parameters for PM3 were derived by comparing a much larger number and a wide variety of experimental versus computed molecular properties. The PM3 is a reparametrization of AM1, which is based on the neglect of diatomic differential overlap (NDDO) approximation. NDDO retains all one-center

differential overlap terms when Coulomb and exchange integrals are computed. The PM3 method is within the unrestricted Hartree–Fock formalism. Finally, the structure, energy, vibrational frequencies and zero point energy were computed ab initio at the HF-SCF level of theory [23] with the program package Gaussian 94 [33]. For all first to fifth row elements (H–Te) a 3-21G(d) basis set was used [34].

3.1. Disodium telluride (modified procedure)

Into a suspension of 8.5 g (0.370 mol) freshly cut sodium chips in 200 ml tetrahydrofuran was added 23.5 g (0.184 mol) tellurium powder and 0.36 g naphthalene. After stirring for 2 days at 25°C, the initially dark green mixture turned pale brown. The brown solid was filtered and washed twice with 20 ml portions of THF. The resulting powder was dried in vacuum to yield 30.0 g (94%) pale brown Na₂Te. The reaction performed under light exclusion gave similar yields.

3.2. Dicyclohexyl telluride (typical procedure)

Into a solution of 3.21 g (18.4 mmol) Na₂Te in 30 ml absolute ethanol, 6.01 g (36.9 mmol) cyclohexyl bromide was added at 0°C during a period of 30 min. The reaction mixture was stirred for further 30 min at 0°C while the purple color turned to brown. Continuous stirring was achieved for 18 h at 25°C. The sodium bromide was separated by centrifugation and the remaining solution was distilled twice under vacuum to yield 2.25 g (42%) of pure dicyclohexyl telluride as a dark orange liquid (b.p. 97°C/0.01 Torr). MS (EI, 70 eV); *m/e* (%): 422 (5, *M*⁺ + Te), 296 (20, *M*⁺), 340 (5, C₆H₁₁Te₂⁺), 214 (10, C₆H₁₁Te⁺), 83 (100, C₆H₁₁⁺). Elemental analysis (%) C₁₂H₂₂Te [293.94] calc.: C, 49.03; H, 7.56. Found: C, 48.77; H, 7.28.

3.3. Di-*n*-propyl telluride

Raman (100 mW) ν in cm⁻¹: 2962 (49), 2921 (100), 2869 (55), 2851 (34), 2727 (7), 1446 (22), 1421 (13), 1323

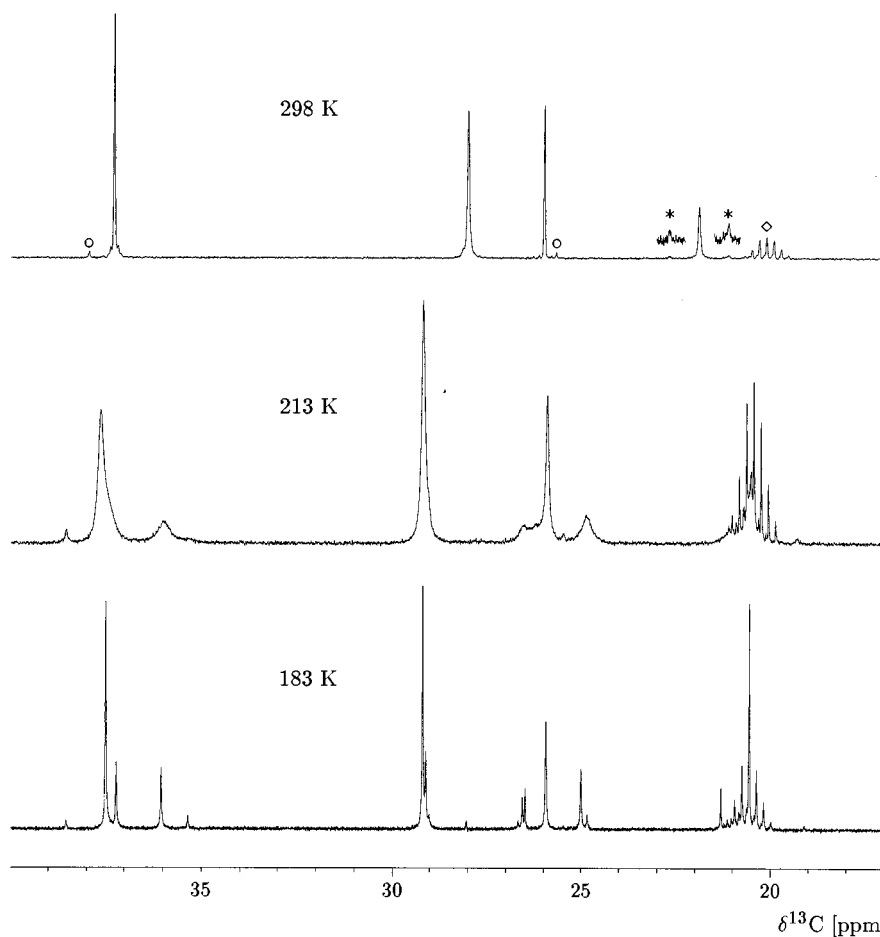


Fig. 3. ¹³C{¹H}-NMR spectra of Te(*cyclo*-C₆H₁₁)₂ at variable temperature in toluene-d₈ (100.54 MHz, ca. 1 M, 300 scans). The ¹²⁵Te satellites of C-1 are marked with (*); ◇ denotes CD₃ resonance of solvent; ○ denotes traces of Te₂(*cyclo*-C₆H₁₁)₂.

(11), 1277 (13), 1181 (30), 1068 (8), 1024 (18), 878 (12), 798 (6), 752 (6), 596 (69), 505 (88), 390 (20), 289 (58), 208 (20), 195 (20).

3.4. Diisopropyl telluride

Raman (100 mW) ν in cm^{-1} : 2970 (38), 2925 (100), 2877 (58), 2718 (9), 1456 (26), 1274 (8), 1204 (31), 1150 (23), 1102 (11), 1055 (9), 1009 (7), 960 (5), 883 (26), 509 (70), 487 (32), 403 (13), 259 (29), 244 (28), 107 (19).

3.5. Di-*n*-butyl telluride

Raman (100 mW) ν in cm^{-1} : 2961 (53), 2919 (100), 2874 (77), 2859 (61), 2735 (9), 1445 (25), 1425 (13), 1301 (12), 1286 (14), 1249 (12), 1183 (23), 1170 (20), 1087 (9), 1051 (12), 1009 (8), 887 (16), 857 (13), 599 (55), 506 (50), 450 (12), 409 (13), 252 (33), 212 (20).

3.6. Di-*n*-hexyl telluride

Raman (100 mW) ν in cm^{-1} : 2960 (40), 2919 (100), 2874 (74), 2859 (67), 2730 (7), 1439 (26), 1304 (16), 1220 (12), 1170 (16), 1063 (8), 964 (7), 843 (10), 776 (6), 603 (41), 507 (40), 480 (15), 434 (9), 402 (7), 379 (8), 330 (6), 236 (17), 195 (21), 184 (19), 172 (19), 118 (15).

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References

- [1] F. Wöhler, Liebigs Ann. Chem. 35 (1840) 111.
- [2] E.G. Hope, T. Kemmitt, W. Levason, Organometallics 7 (1988) 78.
- [3] A. Osuka, H. Suzuki, Tetrahedron Lett. 46 (1983) 5109.
- [4] H. Suzuki, Y. Hanazaki, H. Manabe, R. Eniokiya, Chem. Lett. (1986) 549.
- [5] T. Murai, K. Imaeda, S. Kajita, K. Kimura, H. Ishihara, S. Kato, Phosphorus Sulfur Silicon 67 (1991) 239.
- [6] L. Engman, Acc. Chem. Res. 18 (1985) 274.
- [7] F.F. Knapp Jr., K.R. Ambrose, A.P. Callaghan, J. Nucl. Med. 21 (1980) 251.
- [8] J.B. Mullin, J.C. Irvine, D.J. Ashen, J. Cryst. Growth 55 (1981) 92.
- [9] W.E. Hoke, P.J. Lemonias, Appl. Phys. Lett. 46 (1985) 398.

- [10] D. Klamann, K.J. Irgolic, Houben-Weyl, Methoden der Org. Chemie, 4. Edition, vol. E12b, Organotellurium Compounds, Georg Thieme Verlag, Stuttgart–New York, 1990.
- [11] G. Süß-Fink, M. Langenbahn, H. Stoeckli-Evans, D. Naumann, J. Chem. Soc. Chem. Commun. (1991) 447.
- [12] Y.D. Kulkarni, S. Srivastava, Ind. J. Chem. 24 (1985) 429.
- [13] M.G.M. van der Vis, E.H.P. Cordfunke, R.J.M. Konings, A. Oskam, J. Chem. Soc. Faraday Trans. (1996) 973.
- [14] M.J. Almond, C.A. Yates, D.A. Rice, P.J. Hendra, P.T. Brain, J. Mol. Struct. 239 (1990) 69.
- [15] K.T. Higa, D.C. Harris, Organometallics 8 (1989) 1674.
- [16] J.B. Foresman, A. Frisch, Exploring Chemistry with Electronic Structure Methods, 2nd edn., Gaussian, Pittsburgh, PA, 1993.
- [17] L. Tschugaeff, W. Chlopin, Ber. Dtsch. Chem. Ges. 47 (1914) 1274.
- [18] M.L. Bird, F. Challenger, J. Chem. Soc. (1939) 165.
- [19] K.-H. Thiele, A. Steinicke, U. Dümichen, B. Neumüller, Z. Anorg. Allg. Chem. 622 (1996) 231.
- [20] K.K. Bhasin, V. Gupta, A. Gautam, R.P. Sharma, Synth. Commun. 20 (1990) 2191.
- [21] R. Kasemann, C. Lichenheim, G. Nowicki, D. Naumann, Z. Anorg. Allg. Chem. 621 (1995) 213.
- [22] D.H. O'Brien, N. Dereu, C.-K. Huang, K.J. Irgolic, F.F. Knapp, Organometallics 2 (1983) 305.
- [23] T.M. Klapötke, A. Schulz, R.D. Harcourt, Quantum Chemical Methods in Main-Group Chemistry, Wiley, Chichester, 1998.
- [24] H. Duddeck, P. Wagner, S. Gegner, Tetrahedron Lett. 26 (1985) 1205.
- [25] H. Eggert, O. Nielsen, L. Henriksen, J. Am. Chem. Soc. 108 (1986) 1725.
- [26] H. Duddeck, P. Wagner, A. Biallaß, Magn. Reson. Chem. 29 (1991) 248.
- [27] H. Duddeck, A. Biallaß, Magn. Reson. Chem. 32 (1994) 303.
- [28] (a) D.S. Stephenson, G. Binsch, J. Magn. Reson. 32 (1978) 145. (b) D.S. Stephenson, Program DNMR5, University of Munich, 1994.
- [29] M. Oki, Applications of Dynamic NMR Spectroscopy to Organic Chemistry, VCH Publishers, Deerfield Beach, 1985.
- [30] HyperChem 5.0, Molecular Visualization and Simulation Program Package, Hypercube, Gainesville, FL, 1997.
- [31] (a) J.J.P. Stewart, J. Comp. Chem. 10 (1989) 209. (b) J.J.P. Stewart, J. Comp. Chem. 10 (1989) 221.
- [32] (a) M.J.S. Dewar, W. Thiel, J. Am. Chem. Soc. 99 (1977) 4499. (b) M.J.S. Dewar, M.L. McKee, H.S. Rzepa, J. Am. Chem. Soc. 100 (1978) 3607. (c) M.J.S. Dewar, E.G. Zebisch, E.F. Healy, J. Am. Chem. Soc. 107 (1985) 3902. (d) M.J.S. Dewar, C.H. Reynolds, J. Comp. Chem. 2 (1986) 140.
- [33] Gaussian 94, Revision B.2, M.J. Frisch, G.W. Trucks, H.B. Schlegel, P.M.W. Gill, B.G. Johnson, M.A. Robb, J.A. Cheeseman, T. Keith, G.A. Petersson, J.A. Montgomery, K. Raghavachari, M.A. Al-Laham, V.G. Zakrzewski, J.V. Ortiz, J.B. Foresman, C.Y. Peng, P.Y. Ayala, W. Chen, M.W. Wong, J.L. Andres, E.S. Replogle, R. Gomperts, R.L. Martin, D.J. Fox, J.S. Binkley, D.J. Defrees, J. Baker, J.J.P. Stewart, M. Head-Gordon, C. Gonzalez, J.A. Pople, Gaussian, Inc., Pittsburgh, PA, 1995.
- [34] S. Huzinaga, J. Andzelm, M. Klobukowski, E. Radzio-Andzelm, Y. Sakai, H. Tatewaki, Gaussian Basis Sets for Molecular Calculations, Elsevier, Amsterdam, 1984.