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A TELLURIUM-125 STUDY OF LITHIUM ALKANE- AND ARENETELLUROLATES

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Abstract Lithium alkanetellurolates and lithium arenetellurolates were prepared by reduction of ditellurides with lithium in THF. Tellurium-125 chemical shifts of lithium alkanetellurolates, RTeLi ($\text{R} = \text{Me}, \text{Et}, \text{n-Pr}, \text{i-Pr}, \text{n-Bu}, \text{i-Bu}, \text{s-Bu}$ and t-Bu) and lithium arenetellurolates, $\text{R}'\text{-C}_6\text{H}_5\text{-TeLi}$ ($\text{R}' = \text{para-H}, \text{Me}_2\text{N}, \text{MeO}, \text{Me}_2\text{CHO}, \text{Me}, \text{C}_6\text{H}_5\text{O}, \text{F}, \text{Cl}, \text{Br}, \text{Me}_3\text{Si}$; $\text{R}' = \text{meta-MeO}, \text{EtO}, \text{Me}_2\text{N}, \text{Me}, \text{F}$; $\text{R}' = \text{ortho-MeO}$ and Me_2N) were determined. Chemical shift trends for alkanetellurolates are compared with those previously reported for dialkyl ditellurides, dialkyl tellurides and bis(alkyltelluro)methanes. The ^{125}Te shifts of alkanetellurolates plotted versus ^{77}Se shifts of alkaneselenolates gave an excellent linear correlation. The substituent effects for para- and meta-substituted arenetellurolates are correlated with single- and dual-substituent parameter equations. Single parameter equations gave a poor fit, whereas dual-substituent parameter equations produced good linear correlations. The dual-substituent parameter equation of Taft is slightly better than the two-parameter equation of Swain and Lupton in correlating ^{125}Te shifts of substituted arenetellurolates.

INTRODUCTION

Although interest in tellurium chemistry and tellurium-125 NMR¹ has increased during the past decade, the ^{125}Te shift of -287 ppm for tris(trimethylsilyl)methanetellurolate² is the only ^{125}Te shift for tellurolates reported thus far. Alkane- and arenetellurolates are often used in situ as reagents in substitution reactions,³ in addition reactions to alkynes,^{4,5} in reductive dehalogenation of α -halocarbonyl compounds,⁶⁻⁸ and for the synthesis of symmetric ditellurides.^{2,9-14} The sensitivity of tellurolates toward oxidation makes it difficult to isolate them in pure form and to characterize

them. In this paper we report ^{125}Te chemical shifts for lithium alkanetellurolates and for ortho-, meta- and para-substituted lithium arenetellurolates.

EXPERIMENTAL

Dimethyl ditelluride, di-*t*-butyl ditelluride¹³ and other dialkyl ditellurides were prepared according to literature procedures. Bis(2-dimethylaminophenyl) ditelluride was obtained from 2-dimethylaminophenyllithium¹⁵ and elemental tellurium followed by oxidation of the arenetellurolate.¹³ Diphenyl ditelluride, bis(4-methylphenyl) ditelluride, bis(4-fluorophenyl) ditelluride, bis(4-bromophenyl) ditelluride, bis(4-methoxyphenyl) ditelluride,⁹ bis(4-phenoxyphenyl) ditelluride, bis(4-methoxyphenyl) ditelluride,¹⁶ bis(4-dimethylaminophenyl) ditelluride, bis(4-isopropoxyphenyl) ditelluride, bis(4-trimethylsilylphenyl) ditelluride, bis(4-chlorophenyl) ditelluride, bis(3-methoxyphenyl) ditelluride, bis(3-dimethylaminophenyl) ditelluride, bis(3-methylphenyl) ditelluride, bis(3-ethoxyphenyl) ditelluride, and bis(2-methoxyphenyl) ditelluride¹⁷ were prepared according to literature procedures. The purity of the dialkyl ditellurides and diaryl ditellurides was checked by proton and ^{125}Te NMR.

Preparation of Lithium Alkanetellurolates: The liquid dialkyl ditelluride (0.5 mL) was mixed with dry, oxygen-free THF (3 mL) in an NMR tube under an atmosphere of purified argon. An excess of freshly-cut lithium foil pieces (0.1 g) was added. The tube was stoppered and the mixture shaken vigorously. The reaction started immediately. Slight warming first intensified the color of the red mixture. The color subsequently faded to produce a colorless to yellow solution approximately 0.2 M in alkanetellurolate. Typically, the reactions were completed within one hour.

Preparation of Lithium Arenetellurolates: The solid diaryl ditelluride (0.6 mmol) was weighed into an NMR tube. The tube was evacuated and purged with dry, oxygen-free argon. Dry, oxygen-free THF (3 mL) was then added followed by an excess of freshly-cut,

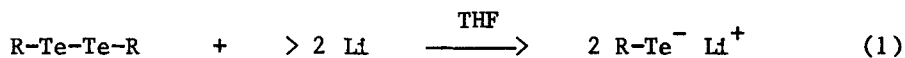
clean lithium foil pieces (0.1 g). The tube was stoppered and shaken vigorously at room temperature until the color had changed from red to yellow. The halogen-substituted diaryl ditellurides had to be shaken for several hours to complete the reduction. The arenetellurolate solutions obtained in this manner were about 0.4 M.

NMR Measurements: All ^{125}Te spectra were obtained with a Varian Associates FT-80 NMR Spectrometer equipped with a broad-band probe tuned to 25.104 MHz operating at ambient temperature. About 30,000 scans were required to obtain satisfactory signal-to-noise ratios using spectral widths of 25 kHz stored in 24K data points. With these spectral parameters, proton noise decoupled tellurium resonances were 30 to 40 Hz wide at half height. The ^{125}Te shifts are reported relative to neat dimethyl telluride (external). Positive shifts are downfield and negative shifts are upfield from dimethyl telluride.

RESULTS AND DISCUSSION

Synthesis of Lithium Tellurolates

Tellurolates needed for synthetic purposes are commonly prepared by insertion of tellurium into a carbon-metal bond.^{2,14,18,19} Tellurolate solutions prepared in this way often contain other tellurium species because carbanions can react with more than one equivalent of tellurium with the formation of polytellurolates, R-Te_n^- .⁶ The presence of tellurolate, R-Te^- , and ditellurolate, R-Te-Te^- , in solution may lead to tellurium exchange and broadening of the ^{125}Te signal. To ensure the exclusive formation of tellurolates, R-Te^- , symmetric diorganyl ditellurides were reduced to tellurolates with



$\text{R} = \text{Me}, \text{Et}, \text{n-Pr}, \text{i-Pr}, \text{n-Bu}, \text{i-Bu}, \text{s-Bu}, \text{t-Bu}$

$\text{R} = 4\text{-R}'\text{C}_6\text{H}_5$: $\text{R}' = \text{Me}_2\text{N}, \text{MeO}, \text{Me}_2\text{CHO}, \text{PhO}, \text{Me}, \text{F}, \text{Cl}, \text{Br}, \text{Me}_3\text{Si}$

$\text{R} = 3\text{-R}'\text{C}_6\text{H}_5$: $\text{R}' = \text{Me}_2\text{N}, \text{MeO}, \text{EtO}, \text{Me}, \text{F}$

$\text{R} = 2\text{-R}'\text{C}_6\text{H}_5$: $\text{R}' = \text{Me}_2\text{N}, \text{MeO}$

an excess of metallic lithium (Eq. 1).

Arenetellurolates can be prepared by this method only when the substituents in the diaryl ditellurides are not attacked by metallic lithium. Although aryl bromides and aryl chlorides usually react with lithium to form aryl lithium compounds, bis(4-bromophenyl) ditelluride and bis(4-chlorophenyl) ditelluride could be reduced to the corresponding tellurolates without halogen-lithium exchange. However, the halogen atoms in bis(4-iodophenyl) ditelluride and bis(3-bromophenyl) ditelluride reacted with lithium and produced products insoluble in THF. These solutions did not give ^{125}Te signals. Tellurolate solutions suitable for NMR studies could not be obtained from bis(3-nitrophenyl) and bis(4-nitrophenyl) ditellurides.

Tellurium-125 Chemical Shifts for Alkanetellurolates

The ^{125}Te chemical shifts of lithium alkanetellurolates prepared according to Eq. 1 are listed in Table I. Compared to other classes

TABLE I ^{125}Te Shifts for Lithium Alkanetellurolates.

$\frac{\text{R-Te-Li}}{\text{R}}$	$\delta_{^{125}\text{Te}}$	$\frac{\text{R-Te-Li}}{\text{R}}$	$\delta_{^{125}\text{Te}}$
Me	- 725	n-Bu	- 535
Et	- 433	i-Bu	- 582
n-Pr	- 532	s-Bu	- 236
i-Pr	- 148	t-Bu	+ 117

of organic tellurium compounds, the range of 842 ppm for the shifts of alkanetellurolates is very large. Methyl substitution at the α -carbon of the alkyl group deshields the tellurium in dialkyl ditellurides,²⁰ dialkyl tellurides,²¹ and bis(alkyltelluro)methanes²² whereas substitution at the β -carbon shields the tellurium nucleus. These effects can be quantified by α -, β -, and γ -substitution increments.^{20,21} The increments for the lithium alkanetellurolates

are given in Table II. Comparison of the substitution increments of alkanetellurolates with the increments for dialkyl ditellurides,²⁰ dialkyl tellurides,²¹ and bis(alkyltelluro)methanes²² shows the

TABLE II ^{125}Te Shift Changes for Alkanetellurolates
Caused by Methyl Substitution.

Substitution ^a	Substitution Increment	Comparison
α_1	+ 292	Me to Et
α_{2-1}	+ 285	Et to <i>i</i> -Pr
α_{2-2}	+ 296	<i>n</i> -Pr to <i>s</i> -Bu
α_3	+ 265	<i>i</i> -Pr to <i>t</i> -Bu
β_1	- 99	Et to <i>n</i> -Pr
β_{2-1}	- 50	<i>n</i> -Pr to <i>i</i> -Bu
β_{2-2}	- 88	<i>i</i> -Pr to <i>s</i> -Bu
γ	- 3	<i>n</i> -Pr to <i>n</i> -Bu

^a Symbols defined in ref. 20.

α -substitution increments of tellurolates to be approximately 100 ppm larger and the β -substitution increments to be equal. The difference between the α -substituent increments can be attributed to the greater asymmetry of the valence electron cloud¹ on tellurium in alkanetellurolates than in dialkyl ditellurides, dialkyl tellurides and bis(alkyltelluro)methanes.

It has been found that the ^{77}Se and ^{125}Te shifts for structurally similar compounds are influenced by the same factors.²³ Therefore, plots of tellurium versus selenium shifts for analogous compounds give straight lines with slopes between 1.7 and 1.8.^{21,23,24} The ^{125}Te shifts of alkanetellurolates plotted against the ^{77}Se shifts for four sodium alkaneselenolates gave an excellent straight line

$$\delta_{^{125}\text{Te}} = 1.81 \delta_{^{77}\text{Se}} - 141.3 \quad (2)$$

with a slope of 1.81 (Eq. 2; $r = 0.998$) in good agreement with results for other selenium and tellurium compounds.^{21,23} Eq. 2 was

used to calculate ^{77}Se shifts for the following alkaneselenolates for which experimental values are not yet available: $n\text{-PrSe}^-$, -215 ppm; $n\text{-BuSe}^-$, -217 ppm; $i\text{-BuSe}^-$, -243 ppm; and $s\text{-BuSe}^-$, -52 ppm.

Tellurium-125 Shifts for Lithium Arenetellurolates

The ^{125}Te chemical shifts for para-, meta-, and ortho-substituted lithium arenetellurolates and their substituent chemical shifts (SCS) are listed in Table III. The ^{125}Te shifts vary in a regular

TABLE III Tellurium-125 Shifts and Substituent Chemical Shifts (SCS) for Lithium Arenetellurolates

$$\text{SCS} = \delta_{^{125}\text{Te}}(\text{RC}_6\text{H}_5\text{TeLi}) - \delta_{^{125}\text{Te}}(\text{C}_6\text{H}_5\text{TeLi})$$

$\text{R-C}_6\text{H}_5\text{TeLi}$	$\delta_{^{125}\text{Te}}$	SCS	$\text{R-C}_6\text{H}_5\text{TeLi}$	$\delta_{^{125}\text{Te}}$	SCS
R			R		
4-Me ₂ N	-210.4	-87.9	3-Me	-151.6	-29.1
4-MeO	-178.5	-56.0	3-Me ₂ N	-127.3	-4.8
4-Me ₂ CHO	-173.1	-50.6	3-MeO	-96.1	+26.4
4-Me	-135.2	-12.7	3-EtO	-96.1	+26.4
4-C ₆ H ₅ O	-133.6	-11.1	3-F	-13.3	+109.2
4-F	-123.9	-1.4			
H	-122.5	0.0	2-MeO	-229.5	-107.0
4-Me ₃ Si	-106.5	+16.0	2-Me ₂ N	-211.1	-88.0
4-Cl	-68.0	+54.5			
4-Br	-63.8	+58.7			

way with the nature of the substituent. Electron-donating groups cause large upfield shift changes and electron-withdrawing groups, large downfield shift changes.

Many different sets of substituent parameters are available in the literature²⁵ to quantitatively correlate field and resonance effects of various substituents with the reactivity or physical properties of a series of compounds. There have been two previous investiga-

tions in which ^{125}Te chemical shifts for dialkyl tellurides²⁶ and for aryl tellurium trichlorides²⁷ are related to substituent constants. Selenium-77 chemical shifts for para-substituted diphenyl selenides²⁸ and for para-substituted phenyl selenobenzoates²⁹ have been related with single- and dual-substituent parameter calculations.

Single- and dual-parameter correlations of ^{125}Te shifts with different substituent parameters for para- and meta-substituted arenetellurolates are summarized in Tables IV and V. Single-parameter, linear regression analyses with Hammett σ -values³⁰ and the σ_p^+ -values of Brown and Okamoto^{31,32} give equations with large standard deviations (SD) and low correlation coefficients. Davis and Pryor have shown that the correlation coefficient is very dependent upon the magnitude of the slope.³³ Therefore, we have included f-values as measures of goodness-of-fit (Eq. 3). An f-value between 0.0 and 0.1 indicates an excellent correlation, values between 0.1 and 0.2,

$$f = \frac{SD}{RMS} \quad \begin{array}{l} SD = \text{standard deviation} \\ RMS = \text{root mean square, SCS values} \end{array} \quad (3)$$

a good correlation, and values greater than 0.3, only trends. The f-values for the single-substituent parameter equations indicate only very crude trends. The single-parameter equations in Tables IV and V are separated into equations for para- and meta-substituents for ease of comparison to the dual-substituent parameter equations. However, the single-parameter Hammett σ correlation is not

$$SCS = 151.4 + 16.9 \quad \begin{array}{l} n = 15; SD = 49.6; \\ r = 0.92; f = 1.01 \end{array} \quad (4)$$

substantially improved even when all 15 para- and meta-substituents are used (Eq. 4).

In order to improve the correlations, the two-parameter equation of Swain and Lupton^{30,34} and the dual-substituent parameter treatment of Craik, Brownlee and Taft^{35,36} were used. The Swain treatment

TABLE IV Correlations of Tellurium-125 Substituent Chemical Shifts (SCS) for Para-Substituted Arenetellurulates with Single and Dual Substituent Parameters.^a

Parameter	Equation	^b n	SD	r	f
Single					
σ_p (Hammett)	$SCS = 137.5\sigma_p + 14.5$	10	46.6	0.94	1.03
σ_p^+ (Brown)	$SCS = 72.2\sigma_p^+ + 23.9$	9	47.0	0.91	1.06
Dual					
F, R (Swain)	$SCS = 53.9F + 38.4R + 6.9$	10	20.4	0.93	0.43
σ_I, σ_R^{BA} (Taft)	$SCS = 152.3\sigma_I + 131.9\sigma_R^{BA}$	9	17.7	0.95	0.37
σ_I, σ_R^+ (Taft)	$SCS = 118.0\sigma_I + 62.2\sigma_R^+$	9	23.1	0.91	0.49
σ_I, σ_R^0 (Taft)	$SCS = 178.5\sigma_I + 208.5\sigma_R^0$	9	19.2	0.94	0.41
σ_I, σ_R^- (Taft)	$SCS = 246.6\sigma_I + 273.3\sigma_R^-$	8	13.1	0.97	0.26

^a SD = standard deviation; r = correlation coefficient; and f = SD/RMS of SCS values.
^b n = number of SCS values; for the Brown and three Taft equations with n = 9, the SCS for isopropoxy was not used; and for the Taft equation with n = 8, isopropoxy and phenoxy SCS's were not used.

TABLE V Correlations of Tellurium-125 Substituent Chemical Shifts (SCS) for Meta-Substituted Arenetellurolates with Single and Dual Substituent Parameters.^a

Parameter	Equation	n^b	SD	r	f
Single					
σ_m (Hammett)	$SCS = 230.0\sigma_m + 12.1$	5	53.4	0.89	1.03
Dual					
F, R (Swain)	$SCS = 185.2F + 32.0R - 17.0$	6	13.0	0.98	0.24
σ_I, σ_R^{BA} (Taft)	$SCS = 238.2\sigma_I + 35.7\sigma_R^{BA}$	5	18.1	0.98	0.31
σ_I, σ_R^+ (Taft)	$SCS = 223.2\sigma_I + 16.2\sigma_R^+$	5	18.9	0.97	0.32
σ_I, σ_R^o (Taft)	$SCS = 248.7\sigma_I + 59.7\sigma_R^o$	5	15.9	0.98	0.27
σ_I, σ_R^- (Taft)	$SCS = 299.3\sigma_I + 98.2\sigma_R^-$	5	11.7	0.99	0.20

^a SD = standard deviation; r = correlation coefficient; and f = SD/RMS of SCS values.

^b n = number of SCS values; for the Swain equation, the SCS for ethoxy was also used.

divides the substituent constant σ into resonance, R , and field, F , contributions for each substituent (Eq. 5). The calculated values

$$\sigma = fF + rR \quad (5)$$

of the weighting factors, f and r , estimate the importance of field and resonance effects for the substituent chemical shifts. The Swain-Lupton treatment of the arenetellurolate data gives a fairly good linear correlation for the para- and meta-substituents (Tables IV and V). The f -values (Eq. 3) are substantially lower than for the single-parameter equations. As expected, the field contribution to the substituent constant for the meta-substituents (85%) is much greater than for the para-substituents (57%).

The dual-substituent parameter treatment of Craik, Brownlee and Taft^{35,36} also divides the substituent constant into field and resonance contributions. For each substituent, a σ_I -value estimates the polar contribution and one of four different σ_R -values, the resonance contribution. Four separate calculations are performed to find the best fit. In order to apply the dual-substituent parameter treatment, a minimal set of substituents is recommended.

The recommended minimal set consists of the following substituents: two different halogens, but not both Cl and Br; H and Me; any two mesomeric withdrawing substituents chosen from CF_3 , CO_2R , $MeCO$, CN , NO_2 ; and any two from Me_2N , NH_2 or MeO . The series of arenetellurolates investigated does not fulfill these conditions. Nevertheless, dual-substituent parameter treatments of para- and meta-substituted arenetellurolates gave good correlations (Tables IV and V). On the basis of the f -values, the dual-substituent parameter treatments of Taft and coworkers are slightly better in correlating

the shifts of para- and meta-arenetellurolates than the Swain-Lupton treatment. For the Taft treatments, the best fit was obtained with the para and meta series by using the σ_R^- -resonance parameters. For the para-substituted arenetellurolates, this

treatment shows that resonance interactions are more important than polar field effects. The best fit for the SCS data for meta-substituted arenetellurolates is obtained with the equation that shows the largest resonance coefficient (25%). This result suggests that resonance contributions are also important in describing tellurium-125 shifts of meta-substituted arenetellurolates.

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