

Bis(phenylethynyl) Telluride as a Tellurium(II) Equivalent. Synthesis of Symmetrical and Unsymmetrical Diaryl Tellurides

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Summary: Bis(phenylethynyl) telluride was found to react readily with 2 equiv of a variety of aryllithium reagents in tetrahydrofuran at $-78\text{ }^{\circ}\text{C}$ to afford diaryl tellurides in excellent yields. As judged by ^{125}Te NMR spectroscopy in tetrahydrofuran at $-105\text{ }^{\circ}\text{C}$, neither a tellurium ate complex nor an unsymmetrical telluride, aryl phenylethynyl telluride, was a detectable intermediate in the reaction. The phenylethynyl group of aryl phenylethynyl tellurides was similarly shown to be substituted by treatment with various aryllithiums to afford unsymmetrical diaryl tellurides.

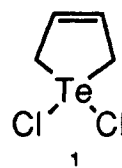
Introduction

As compared with other classes of organotellurium compounds, diaryl tellurides are probably the least sensitive to decomposition by heat, light, or atmospheric oxygen. Despite this stability, the intrinsic weakness of the carbon-tellurium bond, also in these materials, has formed the basis for synthetic applications of the compounds.¹

Many methods are available for the preparation of diaryl tellurides.² Among synthetic methods with some generality, the reaction of telluride ion with aryl halides³ or aryldiazonium compounds,⁴ the addition of organometallic reagents to aryltellurenyl halides,⁵ the copper-induced tellurium extrusion from diaryl ditellurides,⁶ and the reaction of tellurium tetrachloride with aromatic compounds, followed by reduction of the resulting diaryltellurium dihalide,⁷ seem to be the most preparatively useful.

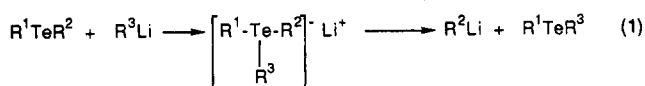
In contrast to organosulfur chemistry, dihalides of the element are not stable with selenium and tellurium.⁸ The products isolated from reactions of "tellurium dihalides" with organolithium and Grignard reagents in older⁹ and recent¹⁰ literature probably resulted from disproportion-

ation products of the dihalide (tellurium and tellurium tetrahalide). When it is treated with at least 4 equiv of an organometallic reagent, tellurium tetrachloride often behaves as a tellurium(II) equivalent, affording a diorganyl telluride.⁸ However, the product is frequently contaminated by the corresponding diorganyl ditelluride and other products, and yields are unsatisfactory. We reported some time ago the use of 2,5-dihydro-tellurophene 1,1-dichloride (1) as a tellurium(II) equivalent.¹¹ In the presence of 2-3



equiv of a Grignard or organolithium reagent, compound 1 afforded diaryl tellurides in fair yields together with butadiene.

Recently, diorganyl tellurides have found extensive use, via lithium/tellurium exchange, in the mild preparation of organolithium compounds. In these reactions, the diorganyl telluride R^1TeR^2 was treated with an organolithium reagent R^3Li to form a tellurium ate complex,¹² which released a thermodynamically more stable organolithium reagent (R^2Li in the example; eq 1). In this way, alkyl-, alkenyl-, alkynyl-, aryl-, acyl-, and carbamoyllithiums were efficiently prepared.¹³⁻¹⁸



We thought it should be possible to prepare symmetrical and unsymmetrical diaryl tellurides via lithium/tellurium exchanges, as outlined in eqs 2 and 3. In the following, we report the successful use of bis(phenylethynyl) telluride

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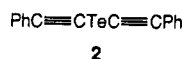
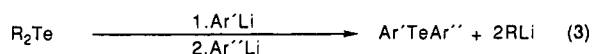
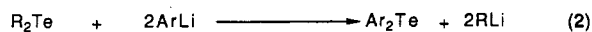
(15) Hiroy, T.; Mogami, T.; Kambe, N.; Fujiwara, S.-I.; Sonoda, N. *Synth. Commun.* 1990, 20, 703.

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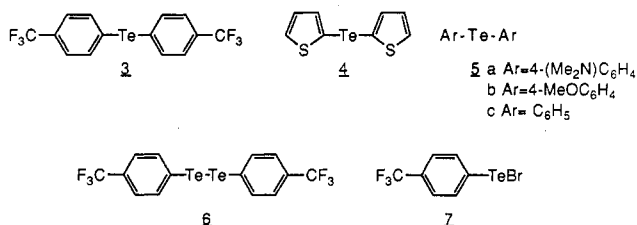
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(2) and other tellurium(II) equivalents for the preparation of diaryl tellurides.



Results

The driving force for the exchange reaction of organolithium compounds, via tellurium ate complexes (eq 1), is the release of a thermodynamically more stable organolithium compound. Therefore, in order to avoid an equilibrium situation where mixtures of diaryl tellurides would result, it is desirable that a Te(II) equivalent be designed to release an organolithium reagent considerably more stable than the one it is going to be reacted with. Since the thermodynamic stability of an organolithium reagent increases with decreasing basicity, diaryl tellurides 3 and 4, which are capable of releasing considerably less basic organolithium reagents than phenyllithium, were initially tried as tellurium(II) equivalents.



When compound 3 was allowed to react in dry tetrahydrofuran at -78°C with 2.5 equiv of [4-(dimethylamino)phenyl]lithium, bis[4-(dimethylamino)phenyl] telluride (5a) was isolated in 34% yield after aqueous workup. When telluride 4 was similarly treated, telluride 5a was isolated in 53% yield. In the presence of 2.5 equiv of (4-methoxyphenyl)lithium, bis(4-methoxyphenyl) telluride (5b) was prepared in 75% yield from telluride 3. When it was treated with (4-methoxyphenyl)lithium (2.2 equiv) and phenyllithium (4.5 equiv), bis(2-thienyl) telluride (4) afforded tellurides 5b and 5c in 85 and 71% yields, respectively.

Telluride 3 is most conveniently prepared by Cu(0)-induced detelluration of its corresponding ditelluride,¹⁹ bis[4-(trifluoromethyl)phenyl] ditelluride (6). Since tellurenyl halides are known to react readily with organometallic compounds, we also investigated the use of tellurenyl bromide 7 as a tellurium(II) equivalent. Addition of 2 equiv of phenyllithium to a solution of in situ prepared (from ditelluride 6 and 1 equiv of bromine) tellurenyl bromide 7 afforded diphenyl telluride (5c) in 58% isolated yield. Similarly, telluride 5a was prepared in 70% yield using 2 equiv of [4-(dimethylamino)phenyl]lithium.

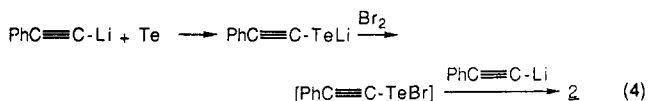
In order to improve the yields and avoid large excesses of the aryllithium reagents, a tellurium(II) equivalent capable of releasing an organolithium reagent considerably less basic than 2-thienyllithium and [4-(trifluoromethyl)-

Table I. Symmetrical Diaryl Tellurides 5 Prepared from Bis(phenylethynyl) Telluride and Aryllithiums

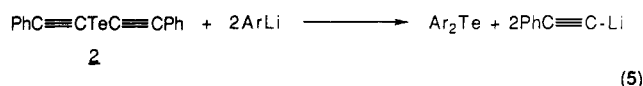
compd no.	Ar	yield ^a (%)	mp ($^\circ\text{C}$)	lit. mp ($^\circ\text{C}$)	ref
5a	4-(dimethylamino)phenyl	92	132	128–130	27
5b	4-methoxyphenyl	100	55	55	7
5c	phenyl	97	160.5 ^b	160	28
5d	4-hydroxyphenyl	96	103	103	19
5e	4-methylphenyl	92	69	69–70	24
5f	3-methylphenyl	100	129 ^b	131–132	30
5g	2,5-dimethylphenyl	86	72	72	31
5h	2,4,6-trimethylphenyl	100	129	129	29
5i	2-thianaphthenyl	78	166.5		c
5j	2-thienyl	75	49	49–50	11
5k	4-(trifluoromethyl)phenyl	82	52	52–53	19

^a Isolated yields. ^b Melting point of Te,Te dichloride. ^c New compound.

phenyl]lithium was looked for. It occurred to us that the known,^{20,21} crystalline, and readily prepared telluride 2, with the potential of releasing (phenylethynyl)lithium, would be ideal for this purpose. Telluride 2 was previously prepared in high yield from phenylethynyl bromide and lithium benzenetelluroate.²⁰ However, due to the sensitivity of phenylethynyl bromide toward polymerization, we found it more convenient to prepare the material from (phenylethynyl)tellurenyl bromide and (phenylethynyl)lithium, as shown in eq 4.

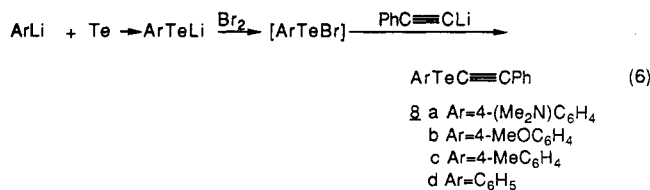


Bis(phenylethynyl) telluride (2) was added to a solution of 2.2 equiv of a variety of aryllithium reagents at -78°C in dry tetrahydrofuran to give symmetrical diaryl tellurides 5 in excellent yields (eq 5; Table I).



The preparation of unsymmetrical diaryl tellurides was also attempted by the consecutive addition of two different aryllithium reagents to telluride 2, as outlined in eq 3. However, these reactions usually produced complex mixtures, where the desired unsymmetrical diaryl tellurides were minor components.

In order to demonstrate the preparation of unsymmetrical diaryl tellurides via lithium/tellurium exchange, a few aryl phenylethynyl tellurides 8 were prepared from aryltellurenyl bromides and (phenylethynyl)lithium in 63–85% yield, as described in eq 6. When some of these



compounds were allowed to react at -78°C in dry THF with 1.5 equiv of a variety of aryllithium reagents,

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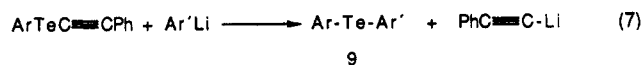
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Table II. Unsymmetrical Diaryl Tellurides 9 Prepared from Aryl Phenylethynyl Tellurides and Aryllithiums

compd no.	Ar	Ar'	yield (%)	mp (°C)	lit. mp (°C)	ref
9a	4-(dimethyl-amino)phenyl	4-methoxy-phenyl	63	97.5	96-97	26
9b	4-(dimethyl-amino)phenyl	phenyl	85	49	49-50	26
9c	4-(dimethyl-amino)phenyl	4-methylphenyl	77	68	70	26
9d	4-(dimethyl-amino)phenyl	4-fluorophenyl	67	55	55	32
9e	4-methoxyphenyl	4-trifluoro-methylphenyl	74	88		b
9f	4-methoxyphenyl	4-methylphenyl	58	69	69	33
9g	4-methoxyphenyl	phenyl	44	63.5	60-61	5

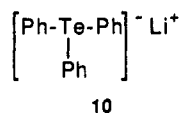
^a Isolated yields. ^b New compound.

unsymmetrical diaryl tellurides 9 were isolated in fair to good yields (eq 7; Table II).



Discussion

As shown by ¹H, ¹³C, ⁷Li and ¹²⁵Te NMR spectroscopy, the tellurium ate complex 10 was quantitatively formed when equal amounts of diphenyl telluride and phenyllithium were added together in dry tetrahydrofuran at -105 °C.¹² Concerning the structure of the complex, ¹H



and ¹³C NMR spectroscopy indicated a T-shaped arrangement of the three phenyl groups around the central atom with two shorter and one longer carbon-tellurium bond. It is likely that the exchange reactions shown in eqs 5 and 7 also occur via tellurium ate complexes. However, an S_N2-like mechanism must also be considered for the reaction, as proposed by Comasseto and co-workers for the closely related substitution of the phenylethynyl group of alkyl phenylethynyl tellurides on treatment with various organometallic reagents.²² In order to distinguish between the two mechanisms, the reaction of telluride 2 with phenyllithium was studied by ¹²⁵Te NMR spectroscopy in tetrahydrofuran at -105 °C. Addition of 0.5 equiv of the organolithium reagent caused a broadening of the peak at 353 ppm (corresponding to telluride 2) and the appearance of a new peak at 670 ppm, corresponding to diphenyl telluride. Addition of another 0.5 equiv of phenyllithium caused the disappearance of the peak corresponding to telluride 2. However, there was no evidence found for the existence of a tellurium ate complex (which could be expected to resonate at higher field than the telluride 2) or for the formation of phenyl phenylethynyl telluride (8d) (resonating at 481 ppm). On the basis of these results, we conclude that the phenylethynyl group is very readily displaced from both bis(phenylethynyl) telluride (2) and the aryl phenylethynyl tellurides 8. If an ate complex were to be formed in the exchange reaction, it is likely to be so close in energy to the transition state that it would be meaningless to classify it as a discrete intermediate.

The high reactivity of the unsymmetrical tellurides 8 toward substitution would also explain the failure to prepare unsymmetrical tellurides, according to eq 3.

As compared with other methods for the preparation of symmetrical diaryl tellurides, the procedure described here in, based on the facile and efficient substitution of the phenylethynyl groups of bis(phenylethynyl)telluride by aryl groups, should be highly attractive. The starting material is readily available, and the high-yield reaction can be carried out under mild conditions, using a variety of aryllithium reagents.

Experimental Part

¹H NMR spectra were recorded on a Bruker AC-F250 instrument at 250 MHz in deuteriochloroform with TMS as an internal standard. ¹²⁵Te NMR spectra were obtained with a Bruker AM 400 instrument operating at 126.242 MHz with a spectral width of 125 kHz. The solutions were 0.13 M in tetrahydrofuran (not deuterated). Chemical shifts are reported in ppm relative to an external reference of neat dimethyl telluride at ambient temperature. Melting points were recorded on a Büchi SMP-20 melting point apparatus and are uncorrected. All aryl bromides were commercially available. All experiments were performed under a positive pressure of Ar. THF was freshly distilled from sodium/benzophenone. Elemental analyses were performed by Analytical Laboratories, Engelskirchen, Germany.

Bis[4-(trifluoromethyl)phenyl] ditelluride was prepared according to a literature procedure.¹⁹ Bis(2-thienyl) telluride (mp 49 °C (lit.¹¹ mp 49-50 °C)) was prepared in 96% yield by Cu(0)-induced detelluration⁶ of bis(2-thienyl) ditelluride.²³

Bis(phenylethynyl) Telluride (2). To a stirred solution of phenylacetylene (0.31 g, 3.0 mmol) in THF (10 mL) was added *n*-BuLi (1.2 mL, 2.5 M, 3.0 mmol) dropwise at -78 °C. After 20 min freshly crushed tellurium powder (0.38 g, 3.0 mmol) was added in one lot while a stream of argon was passed through the open flask. The cooling bath was then removed and the temperature allowed to rise to ambient. When almost all the tellurium was consumed, the reaction mixture was cooled to -78 °C, a solution of bromine (0.48 g, 3.0 mmol) in dry benzene (5 mL) was added dropwise, and stirring was continued for 15 min. (Phenylethynyl)lithium, prepared as described above from phenylacetylene (0.31 g, 3.0 mmol) and *n*-BuLi (1.2 mL, 2.5 M, 3.0 mmol) in THF (10 mL), was then added dropwise at -78 °C and stirring continued for 30 min. The reaction mixture was hydrolyzed at -78 °C by addition of water (5 mL). Dilution with water (20 mL) at ambient temperature, extraction with methylene chloride (2 × 20 mL), drying (MgSO₄), and flash chromatography (1/4 methylene chloride/hexanes) afforded 0.78 g (79% yield) of the title compound as yellow crystals, mp 82 °C (lit.²⁰ mp 83-84 °C).

Syntheses of Symmetrical Diaryl Tellurides 5: Typical Procedure. Bis(4-methylphenyl) Telluride (5e). To a stirred solution of 4-bromotoluene (0.57 g, 3.34 mmol) in THF (10 mL), was added *t*-BuLi (3.9 mL, 1.7 M, 6.69 mmol) dropwise at -78 °C. After 30 min, a solution of bis(phenylethynyl) telluride (0.5 g, 1.52 mmol) in THF (5 mL) was added dropwise at -78 °C and the reaction mixture was kept at -78 °C for an additional 30 min. Hydrolysis at -78 °C, the usual workup as described above, and flash chromatography (1/9 methylene chloride/hexanes) afforded 0.43 g (92% yield) of the title compound, mp 69 °C (hexanes) (lit.²⁴ mp 69-70 °C).

5i: ¹H NMR δ 7.71-7.79 (several peaks, 3H), 7.25-7.33 (several peaks, 2H). Anal. Calcd for C₁₆H₁₀S₂Te: C, 48.78; H, 2.56. Found: C, 48.63; H, 2.50.

For yields and melting points of diaryl tellurides, see Table I. In order to compare the melting points with literature data, the oily diaryl tellurides 5c and 5f were converted to their

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corresponding crystalline Te₂Te dichlorides by addition of a stoichiometric amount of SO₂Cl₂ to a CH₂Cl₂/hexanes solution of the tellurides at 0 °C.

In the preparation of compound **5d**, 3 equiv of *t*-BuLi was added to a stirred solution of 4-bromophenol.

Diaryl tellurides **3** and **4** were added to solutions of aryllithium reagents (2.2–4.5 equiv; see text) as described in the typical procedure for the syntheses of symmetrical diaryl tellurides.

Tellurenyl bromide **7** was generated in situ by the addition of Br₂ (0.22 g, 1.35 mmol) in benzene (10 mL) to a solution of bis-[4-(trifluoromethyl)phenyl] ditelluride (0.74 g, 1.35 mmol) in tetrahydrofuran (15 mL) at -78 °C. After addition of 2 equiv of the aryllithium reagent, workup was carried out as described in the typical procedure for the preparation of symmetrical diaryl tellurides.

Synthesis of Aryl Phenylethynyl Tellurides 8. Typical Procedure. 4-Methoxyphenyl Phenylethynyl Telluride (8b). To a stirred solution of 4-bromoanisole (1.0 g, 5.35 mmol) in THF (20 mL) was added *t*-BuLi (6.3 mL, 1.7 M, 10.7 mmol) at -78 °C. After 15 min, finely crushed tellurium (0.68 g, 5.35 mmol) was added in one lot, while a stream of argon was passed through the open system. The cooling bath was then removed and the temperature allowed to rise to ambient. When almost all the tellurium was consumed, the reaction flask was cooled to -78 °C again. A solution of bromine (0.86 g, 5.35 mmol) in dry benzene (5 mL) was added dropwise and stirring continued for 15 min. The resulting solution was then added dropwise to (phenylethynyl)lithium, prepared as described above from phenylacetylene (0.55 g, 5.35 mmol) and *n*-BuLi (2.1 mL, 2.5 M, 5.35 mmol) in THF (20 mL) at -78 °C. After it was stirred for 20 min, the reaction mixture was hydrolyzed at -78 °C with workup as described above, including flash chromatography (1/4 methylene chloride/hexanes). A 1.53-g amount (85% yield) of the title compound, mp 75 °C (lit.²² mp 72–73 °C), was obtained. Yields, melting points, and ¹H NMR and analytical data (new compounds only) are presented below.

8a: yield 63%; mp 107.5–108 °C; ¹H NMR δ 7.69 (d, 2H), 7.40 (m, 2H), 7.25–7.29 (several peaks, 3H), 6.63 (d, 2H), 2.96 (s, 6H).

Anal. Calcd for C₁₆H₁₅NTe: C, 55.08; H, 4.33. Found: C, 54.97; H, 4.33.

8c: yield 78%; mp 71 °C (lit.²⁵ mp 74–76 °C).

8d: yield 84%; yellow oil. Spectroscopic data were in good agreement with literature data.²⁵

Synthesis of Unsymmetrical Diaryl Tellurides 9: Typical Procedure. 4-(Dimethylamino)phenyl 4-Methoxyphenyl Telluride (9a). To a stirred solution of 4-(dimethylamino)phenyl phenylethynyl telluride (0.30 g, 0.86 mmol) in THF (10 mL) was added a solution of (4-methoxyphenyl)lithium, prepared from 4-bromoanisole (0.24 g, 1.29 mmol) and *n*-BuLi (0.52 mL, 2.5 M, 1.29 mmol) in THF (25 mL), dropwise at -78 °C. Stirring was continued for 50 min at -78 °C and 1 h at ambient temperature. Workup as described above and flash chromatography (1/1 methylene chloride/hexanes) afforded 0.20 g (63%) of the title compound, mp 97.5 °C (lit.²⁶ mp 96–97 °C).

For yields and melting points of diaryl tellurides **9**, see Table II.

9e: ¹H NMR δ 7.79 (d, 2H), 7.53 (m, 2H), 7.36 (m, 2H), 6.84 (d, 2H), 3.83 (s, 3H). Anal. Calcd for C₁₄H₁₁F₃O₂Te: C, 44.27; H, 2.92. Found: C, 44.01; H, 2.89.

Acknowledgment. Financial support by the Swedish Natural Science Research Council and the National Swedish Board for Technical Development is gratefully acknowledged.

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