

# Preparation of symmetrical divinyl tellurides via an ylidation reaction

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## Abstract

Symmetrical divinyl tellurides are prepared by sequential reaction of bis-(triphenylmethyl phosphonium) halotellurate with base or by the reaction of triphenylmethylenide phosphoranes with TeCl<sub>4</sub>, followed by aldehydes. The products are converted to *E*- $\alpha,\beta$ -unsaturated aldehydes by treatment with *n*-BuLi and DMF. © 1999 Elsevier Science S.A. All rights reserved.

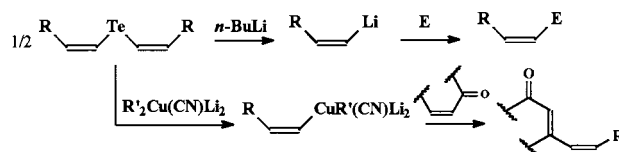
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## 1. Introduction

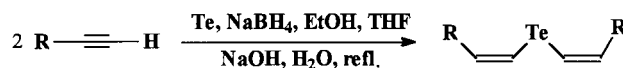
Tellurium compounds have recently attained a remarkable development as synthetic reagents and intermediates [1]. Among the many classes of organotellurium compounds, vinylic tellurides play an important role [2], justifying a recent review concerning their preparation and reactivity [3].

The most important synthetic applications of vinylic tellurides are certainly the detellurative coupling reactions and the transmetalation reaction, i.e. the substitution of the tellurium moiety by the organic moiety of different organocuprates [4], and the Li/Te or Cu/Te exchange giving, respectively, vinyl lithium [5] or vinyl-copper reagents [6].

Symmetrical divinyl tellurides are especially useful in transmetalation reactions since by treating them with two equivalents of Li or copper reagents, both of the vinyl groups are displaced. This process occurs with retention of the carbon–carbon bond geometry and the resulting Li derivative can be trapped by a variety of electrophiles.



A straightforward method for the preparation of symmetrical divinyl tellurides is based on the hydrotelluration of acetylenes with the system Te/NaBH<sub>4</sub>/EtOH, THF/aq NaOH [6a]. However, this method, which produces predominantly *Z* adducts, works well only with acetylenes conjugated to aryl groups, C=C double bonds or other electron-withdrawing groups. By contrast, alkylacetylenes are fairly unreactive, in accordance with the unsatisfactory results described for the addition of organotellurates to these acetylenes [5b,6].



## 2. Results and discussion

New methods for the preparation of symmetrical divinyl tellurides deserve additional efforts. In this paper we describe the preparation of these compounds in

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Yield 0.09 g, 34%. <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>, δ ppm) 0.95–1.12 (m, 12H); 2.2–2.5 (m, 2H); 6.06 (dd, 1H, *J* = 9.2 Hz, *J* = 8.8 Hz); 6.20 (dd, 1H, *J* = 16.2 Hz, *J* = 6.6 Hz); 6.21 (dd, 1H, *J* = 16.2 Hz, *J* = 6.6 Hz); 6.40 (d, 1H, *J* = 8.8 Hz); 6.43 (d, 1H, *J* = 9.2 Hz); 6.48 (dd, 1H, *J* = 16.4 Hz, *J* = 1.2 Hz); 6.49 (dd, 1H, *J* = 16.4 Hz, *J* = 1.2 Hz). MS (*m/z*): 268 [M<sup>+</sup>], 95, 69 (100%), 41.

### 3.3. (*Z,E*)-Phenyl-1-hydroxy-3-phenylprop-2-ene from (*Z,Z*; *E,Z* and *E,E*)-bis-(2-phenyl ethenyl) telluride

To a solution of bis(2-phenylethenyl) telluride (0.334g, 1 mmol) in THF (4 ml) at –78°C under nitrogen, a solution of *n*-BuLi (0.91 ml, 2.2 mmol, 2.42 M solution in hexane) was added dropwise. After 20 min of stirring at this temperature, benzaldehyde (0.265 g, 2.5 mmol) was added. The mixture was stirred for 30 min, then diluted with ethyl acetate (30 ml) and washed with brine (4 × 15 ml). The organic layer was separated, dried over MgSO<sub>4</sub> and solvent removed under vacuum. The residue was purified by column chromatography over silica gel. Elution with hexane removed dibutyl telluride, and elution with ethyl acetate gave an oil. Yield 0.269 g, 64%, *E:Z* = 1:5. <sup>1</sup>H-NMR (200 MHz, CDCl<sub>3</sub>, δ ppm) *E* + *Z* isomers: 2.59 (s, 1H); 7.10–7.40 (m, 10H). *Z* isomer: 5.55 (d, 1H, *J* = 9.2 Hz); 5.85 (dd, 1H, *J* = 11.4, 9.2 Hz); 6.59 (d, 1H, *J* = 11.4 Hz). *E* isomer: 5.25 (d, 1H, *J* = 6.2 Hz); 6.29 (dd, 1H, *J* = 15.8 Hz, *J* = 6.2 Hz); 6.59 (d, 1H, *J* = 15.8 Hz).

### 3.4. (*E*)-*p*-Methylcinnamaldehyde **10b** from (*Z,Z*; *E,Z* and *E,E*)-bis-[2(*p*-tolyl)ethenyl] telluride

To a solution of bis-[2(*p*-tolyl) ethenyl] telluride (0.362 g, 1 mmol) in THF (4 ml) at –78°C under nitrogen, a solution of *n*-BuLi (0.91 ml, 2.2 mmol, 2.42 M solution in hexane) was added dropwise. After 20 min of stirring at this temperature, DMF (0.2 ml, 2.58 mmol) was added. The temperature was raised to room temperature and stirred for 2 h, then diluted with ethyl acetate (30 ml), and washed with water (3 × 20 ml). The organic layer was separated, dried with MgSO<sub>4</sub> and solvent removed under vacuum. Elution with hexane removed dibutyl telluride, and elution with ethyl acetate gave a low m.p. solid, 0.175 g, 60% (characterized as the semicarbazone: m.p. 208.4–209.4°C; Literature [11]

210°C). <sup>1</sup>H-NMR (200 MHz, CDCl<sub>3</sub>, δ ppm) 2.37 (s, 3H); 6.65 (dd, 1H, *J* = 15.9, 7.6 Hz); 7.21 (d, 2H, *J* = 8.0 Hz); 7.37–7.46 (m, 3H); 9.65 (d, 1H, *J* = 7.6 Hz).

### Acknowledgements

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