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### A NOVEL AND CONVENIENT SYNTHESIS FOR THE PREPARATION OF DIALKYL TELLURIDES AND DITELLURIDES

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## A NOVEL AND CONVENIENT SYNTHESIS FOR THE PREPARATION OF DIALKYL TELLURIDES AND DITELLURIDES

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**Abstract** Commercially available hydrazine hydrate rapidly and quantitatively cleaves elemental tellurium,  $\text{Te}_x$ , in strongly basic medium to  $\text{Te}_2^{2-}$ , and to  $\text{Te}^{2-}$  in the presence of sodium dithionite. The reaction proceeds smoothly in water and in all solvents which are unreactive towards strong bases and nucleophiles. Subsequent addition of electrophiles affords dialkyl ditellurides and tellurides in excellent yields.

### INTRODUCTION

The field of organotellurium chemistry has grown rapidly over the last few years as a result of the development of tellurium "reagents" that have unique and subtle applications in organic synthesis<sup>1,2</sup>. As a result, diorganyl tellurides and ditellurides have frequently been used as starting materials and convenient synthetic methods for the synthesis of these compounds are desirable. A useful approach to the synthesis of the title compounds is based on the alkylation of the telluride or ditelluride anion, respectively, which can be prepared from elemental tellurium by reaction with sodium formaldehyde sulfoxylate in aqueous alkali<sup>3</sup>, sodium in liquid ammonia<sup>4,5</sup>, lithium triethyl-borohydride<sup>6</sup> in THF, tetralkyl ammonium borohydride<sup>7</sup> in toluene and alkyl lithium in THF<sup>8</sup>. Recently ultrasonic techniques have also been used for the convenient and selective electrochemical synthesis<sup>9</sup> of the title compounds. We wish to report in this paper a new and convenient method for the preparation of diorganyl ditellurides and diorganyl tellurides.

### EXPERIMENTAL

All reactions are carried out under a nitrogen atmosphere to prevent oxidation of the oxygen-sensitive telluride and ditelluride ions. Tellurium was used in its powdered grey form and stored in a desiccator. The alkyl halides were purified by distillation and were subsequently deoxygenated with a stream of nitrogen. The  $^1\text{H}$  NMR spectra were recorded in  $\text{CCl}_4$ , relative to TMS using a Varian EM 390 operated at 90 MHz.

#### Preparation of dialkyl ditellurides (General Preparation)

Tellurium (6.35 g, 50 mmol) is added to a stirred solution of sodium hydroxide (3.0 g, 75 mmol) in deoxygenated water (20 ml). The mixture is cooled in a water bath, and 100% hydrazine hydrate (2.5 g, 200 mmol) is added over a period of 30 minutes and stirring is continued for an additional hour at room temperature. The alkyl halide (50 mmol) is then added dropwise over a period of 2–3 hours. During the addition, the temperature is maintained at 15–20°C. The end-point of the alkylation is indicated by a sharp color change from dark brown to nearly colorless. The mixture is then extracted with solvent ether. The organic layer is washed with water, dried over anhydrous sodium sulphate and the solvent is removed by slow distillation. The residual product is distilled under reduced pressure (Table 1).

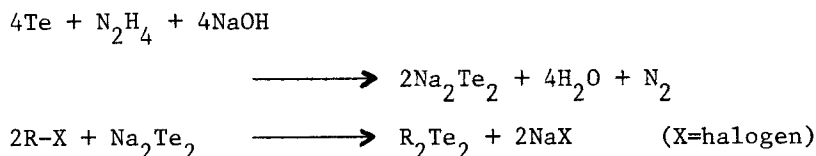
#### Preparation of dialkyl tellurides (General Preparation)

Tellurium (6.35 g, 50 mmol) is added to a stirred solution of sodium hydroxide (6.0 g, 100 mmol) in deoxygenated water (20 ml) cooled in a water bath. To this mixture, 100% hydrazine hydrate (2.5 g, 200 mmol) is added dropwise over a period of 30 minutes and stirring is continued for an additional hour at room temperature. To this dark red solution is added sodium dithionite (23.70g, 125 mmol) portion-wise and stirring is continued until the reaction mixture assumes a heterogeneous off-white color. It

is interesting to note that the color changes during this process. The solution becomes deep blue before turning off-white. During the addition the mixture is continuously stirred and cooled with ice. Following the completion of the reaction the temperature is lowered to  $-10^{\circ}\text{C}$  and alkyl halide (110 mmol) is added over a period of 2-3 hours. The reaction is allowed to stir for another 5-6 hours. A heavy mass separates to the bottom. This is extracted with solvent ether. The organic layer is washed with water, dried over anhydrous sodium sulphate and solvent is removed by slow distillation. The residual product is distilled under reduced pressure (Table 1).

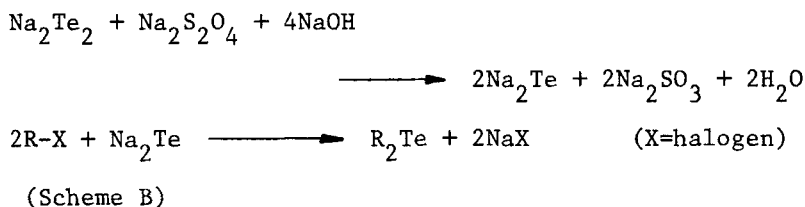
### DISCUSSION

We have recently found that elemental tellurium reacts with an aqueous solution of hydrazine hydrate in the presence of a strong base to give a dark red brown solution containing the ditelluride anion. The reaction is accompanied by a brisk evolution of nitrogen. The resulting solution reacts with alkyl halides to furnish dialkyl ditellurides in almost quantitative yields indicating that a species chemically equivalent to  $\text{Te}_2^{2-}$  is cleanly formed. The reaction proceeds in water and in all solvents unreactive towards strong bases and nucleophiles (Scheme A).



(Scheme A)

Attempts to reduce elemental tellurium to telluride anion,  $\text{Te}^{2-}$  by hydrazine hydrate were unsuccessful. However, the ditelluride anion obtained by reducing tellurium with hydrazine hydrate can be reduced smoothly to the telluride anion by fresh sodium dithionite in the presence of a strong base (Scheme B).



The extension of this method for preparing diaryl analogs is currently under investigations. In view of the inexpensive and readily available starting materials, we feel that the one pot high-yield synthesis reported herein may represent the simplest method currently available.

#### ACKNOWLEDGEMENTS

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