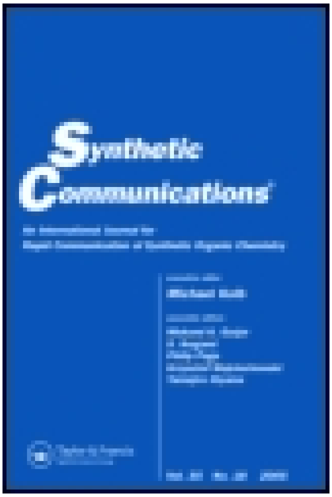

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The Reactions Between Sodium Hydrogen Telluride and Ar_3CX, Ar_2CHX, $ArCH_2X$

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THE REACTIONS BETWEEN SODIUM HYDROGEN TELLURIDE AND Ar_3CX, Ar_2CHX, $ArCH_2X$

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ABSTRACT: Tertiary, secondary and primary benzylic halides give dehalogenated, coupling and oxidized product after reacted with sodium hydrogen telluride in DMF. The yields are 87%, 48% and 40%-56%, respectively.

As a good nucleophile, sodium hydrogen telluride has many useful applications in organic synthesis. Examples are the dealkylation of esters[1] of aromatic acid, ring opening of epoxide,[2] deprotection of chloroethyl carboxylates,[3] N- and/or C-terminal protected α-aminoacids,[4] and the dealkylation of ammonium salts[5,6].

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This article reports the reactions between NaTeH and triphenylmethyl chloride, 9-bromofluorene and several benzyl halides. It is found that 1) NaTeH dechlorinates triphenylmethyl chloride to give triphenylmethane in 87%; 2) NaTeH attacks 9-bromofluorene to afford fluorene and bisfluorene with yields being 33% and 15% respectively; and 3) NaTeH reacts with $ArCH_2X$ to give $ArCH_2TeH$ which in air decomposes to the corresponding aromatic aldehyde (equation 3). The yield is 40%, 50% and 56% respectively for 2-chlorobenzaldehyde, 4-chlorobenzaldehyde and 2-naphthaldehyde. The reaction sequence provides an easy way for synthesizing aromatic aldehydes from the corresponding halides. From the above results, it is clear that NaTeH is a good reagent for dehalogenation of tertiary halide; aromatic aldehydes can be obtained after mild oxidation of organotellurium species which is formed via reaction (3).

$$Ph_3C\text{-}Cl + NaTeH \xrightarrow{DMF} Ph_3C\text{-}H \qquad (1)$$

$$\text{9-bromofluorene} + NaTeH \xrightarrow{DMF} \text{Fluorene} + \text{bisfluorene} \qquad (2)$$

$$ArCH_2X + NaTeH \xrightarrow[\text{2. } CH_3COOEt]{\text{1. DMF}} ArCHO \qquad (3)$$

Experimental Section

Typical procedure for the reaction between NaTeH and halide was given as following:

To 20 mL dry DMF in a 50-ml three-necked round bottom of flask, 1.27 g Te powder and 0.38 $NaBH_4$ were added under a continuous stream of argon . After the stirred mixture was

Table1. The Reactions Between NaTeH And Halides In DMF.

Reactant	Product	mp (°C) or bp	NMR(δ)
Ph_3CCl	Ph_3CH	91-2	1H(s,5.47) 15H(m,7.08-7.30)
9-Br-fluorene	Fluorene	112-4	2H(s,3.87) 8H(m,7.29-7.79)
	Bisfluorene	242-2	2H(s,4.95) 16H(m,7.05-7.77)
2-Cl-$PhCH_2Cl$	2-Cl-PhCHO	215[a]	1H(s,9.97) 4H(m,7.35-7.95)
4-Cl-$PhCH_2Cl$	4-Cl-PhCHO	47	1H(s,9.95) 4H(m,7.5-7.85)
2-$ClCH_2C_{10}H_7$	2-$C_{10}H_7CHO$	58	1H(s,10.14) 7H(m,7.5-8.1)

a. bp (°C).

heated at 70 °C for 30 minutes, a purple solution of sodium hydrogen telluride was obtained. Then, 0.005 mol of halide in 5 mL of DMF was injected in by syringe. The mixture was stirred at the same temperature for hours. After TLC showed that the reaction was complete, the mixture was cooled to room temperature and exposed to air by leaving the flask open. Then 30 mL water was added and solid was filtered off. The filtrate and the solid were extracted with ethyl acetate three times (3X30 mL).[7] The organic layer was washed with saturated NaCl solution twice (2X5 mL). After it was dried over Na_2SO_4 , ethyl acetate was removed in vacuum to give products which were subjected to further separation and purification by chromatography. Yield for triphenylmethane is

87%. For reaction 2, 33% fluorene and 15% bisfluorene were isolated. For reaction 3, the yields ranged from 40% to 56% with aldehyde as product.

Data for products are listed in table 1. Melting points, IR and NMR spectra of products were found identical to those of standards.

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7. For the $ArCH_2X$, the ethyl acetate solution was stirred for 24 hours in air to give aldehyde.

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