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Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/lcyc20>

The Reaction Between Sodium Hydrogen Telluride and Phase Transfer Catalysts

Weixing Li ^{a b} & Xun-Jun Zhou ^a

^a Department of Chemistry , Hangzhou University , Hangzhou, 310028, P. R. CHINA

^b Department of Biomolecular Chemistry , University of Wisconsin , Madison, WI, 53706-1532

Published online: 23 Sep 2006.

To cite this article: Weixing Li & Xun-Jun Zhou (1995) The Reaction Between Sodium Hydrogen Telluride and Phase Transfer Catalysts, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 25:22, 3635-3639, DOI: [10.1080/00397919508015499](https://doi.org/10.1080/00397919508015499)

To link to this article: <http://dx.doi.org/10.1080/00397919508015499>

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THE REACTION BETWEEN SODIUM HYDROGEN TELLURIDE AND
PHASE TRANSFER CATALYSTS

Weixing Li*, Xun-Jun Zhou

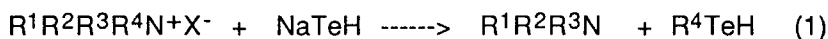
Department of Chemistry, Hangzhou University,
Hangzhou, 310028, P. R. CHINA

ABSTRACT: Phase transfer catalysts reacted with sodium hydrogen telluride with yields ranging from 79% to 94%.

Nucleophilic substitution is one of sodium hydrogen telluride's important characteristics in synthesis. Thus this reagent was employed in ring opening of epoxides,¹ dealkylation of esters of aromatic acids,² bromoethyl carboxylate,³ dehalogenation of dibromo compounds,⁴ and the deprotection of N- and C-protected α -aminoacids.⁵ Most of above reactions are believed to involve two steps, the nucleophilic attack of HTe^- and the decomposition of the tellurium species.

* To whom all correspondence should be addressed. Current address : Department of Biomolecular Chemistry, University of Wisconsin, Madison WI 53706-1532

In this article, we report that, in DMF, sodium hydrogen telluride reacted quickly with benzyl, allyl and methyl containing quaternary ammonium salts, some of which are important phase transfer catalysts, with yields ranging from 79% to 94% (equation 1). It is found that, when methyl and benzyl are attached to the N atom in the ammonium salt, NaTeH attacks preferentially at benzyl (Table 1, entries 1-4). When allyl and ethyl are attached to the N atom, NaTeH reacts with allyl (Table 1, entry 6). Butyl is inert to NaTeH (Table 1, entry 8). Therefore from results listed in table 1, it can be seen that the reaction follow S_N2 mechanism with the order of reactivity being benzyl > methyl, ethyl > other alkyl groups and ally > ethyl.



R⁴ = PhCH₂, allyl, methyl.

R¹, R², R³ please refer to table 1.

The reaction product from R⁴= PhCH₂, namely, ArCH₂TeH, after was stirred in air for about 24 hours in ether or in ethyl acetate, and was found to decompose to give benzaldehyde.

Therefore, our results indicate that quaternary ammonium phase transfer catalysts containing benzyl, allyl and methyl

Table 1. The Reaction Between NaTeH and $R^1R^2R^3R^4N^+X^-$ in DMF

No	Reactant	Product	Yield	mp ^a or bp	mp ^{lit} or bp
1	$\text{PhCH}_2\text{N}^+\text{MePhCl}^-$	PhNMe_2	81%	193-4	193 ⁶
2	$\text{PhCH}_2\text{N}^+(\text{Me})_3\text{PhBr}^-$	PhNMe_2	94%	193-4	193 ⁶
3	$\text{PhCH}_2\text{N}^+(\text{Me})_2\text{CH}(\text{OH})\text{CHPhMe}_2\text{Br}^-$	$\text{Me}_2\text{NCH}(\text{OH})\text{CHPh}$	91%	84-86	87 ⁹
4	$\text{PhCH}_2\text{N}^+(\text{C}_2\text{H}_5)_3\text{Br}^-$	$\text{N}(\text{C}_2\text{H}_5)_3$	91%	88-9	89 ⁷
5	$\text{PhCH}_2\text{N}^+(\text{C}_2\text{H}_5)_3\text{Cl}^-$	$\text{N}(\text{C}_2\text{H}_5)_3$	94%	88-9	89 ⁷
6	$\text{CH}_2=\text{CHCH}_2\text{N}^+\text{Et}_3\text{Br}^-$	$\text{N}(\text{C}_2\text{H}_5)_3$	79%	88	89 ⁷
7	$\text{PhCH}_2\text{N}^+(\text{Me})_2\text{PhBr}^-$	PhCHO	38%	235-6 ^b	237 ^{b,8}
8	$(\text{C}_4\text{H}_9)_4\text{N}^+\text{Cl}^-$	-	-	-	-

a. in °C. b. mp of 2,4-dinitrophenylhydrazone.

are not suitable for NaTeH related PTC reactions because NaTeH will decompose these catalysts.

Experimental Section

Typical procedure for the reaction between NaTeH and phase transfer catalysts was as follows:

1) The reaction between phase transfer catalysts and NaTeH: To 15ml DMF in a 50-ml three necked round bottom flask, 1.27g (0.01 mole) Te powder and 0.38g (0.01 mole) NaBH₄ were added under a continuous stream of nitrogen . After the mixture was heated with stirring at 70°C for 30 min, a purple solution of NaTeH was obtained. Then, at about 40°C, 1.46g benzyldimethylphenylammonium bromide in 5ml DMF was injected in by syringe. The mixture was stirred at this temperature for 4 hours. After TLC showed that the reaction was complete, the mixture was cooled to room temperature and exposed it to air. Then 30 ml water was added and black solid filtered off , which contains the organotellurium species and will be used in 2). The filtrate was extracted with ether three times (3X20ml), the ether layer washed with saturated NaCl solution twice (2X5ml). After it was dried over Na₂SO₄ , ether was removed in vacuum to give a liquid, from which 0.57g N,N-dimethylaniline was obtained after using column chromatography, yield being 94%.

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

2) Decomposition of PhCH₂TeH:

The black solid from above was extracted with 60ml ether. The ether solution was stirred under atmosphere for 24 hours, filtered , and the ether removed in vacuum. The liquid was purified by column chromatography, affording 0.2 g

benzaldehyde, yield being 38%. NMR and 2,4-dinitrophenylhydrazine test were employed to confirm the aldehyde ($\delta=10.01, 1\text{ H}$; $\delta=7.5-7.90, 5\text{ H}$).

Acknowledgement: We were grateful to the National Science Foundation of China for financial support.

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(Received in the USA 12 April 1995)