Chemoselective reduction of $\alpha$, $\beta$-unsaturated carbonyl compounds by sodium hydrogen telluride: Part I

G Geethamalika, A Suguna Sundari, P Shanmugam & S P Rajendran
Department of Chemistry, Bharathiar University, Coimbatore 641 046

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Sodium Hydrogen Telluride is used for the selective reduction of C=C bonds in various chalcones. The reduction proceeds smoothly in good yields.

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Sodium hydrogen telluride has earlier been shown by Shanmugam and co-workers of our department to serve as a convenient reagent for the hydrogenation of carbon-carbon double bonds, for the debromination of vicinal dibromides, for the decacylation of aryl acetates and benzoates, for deblocking allyl carboxylates and allyl phenyl ethers and for the partial deallylation of 3,3-diallyl-1,2,3,4-tetrahydroquinolin-2,4-diones. In continuation of our work on sodium hydrogen telluride, we wish to report herein on the further utility of sodium hydrogen telluride as a mild and effective reagent for the selective reduction of C=C double bond in chalcones.

We have now reduced a few $\alpha$, $\beta$-unsaturated carbonyl compounds la - 8a which also include the newer chalcones viz. 5a - 8a (Table I). All these compounds have been prepared in our laboratory for the further synthesis of heterocycles. The compounds have been reduced by NaHTe prepared in situ from tellurium powder and sodium borohydride on refluxing for 4-5 hr under nitrogen cover. The reduction proceeded smoothly in each case with the product yields varying from 71-95% and without the formation of any side products. This method has an advantage that it is operationally smooth, the conditions are mild and tellurium metal can be recovered almost quantitatively and reused.

Experimental Section
Melting points were determined on a Boetius micro-

Note
heating table/in open capillaries and are uncorrected. IR spectra (KBr) were recorded on a Perkin-Elmer 597 infrared spectrophotometer and NMR spectra on a Hitachi R-600 spectrometer/AMX-400 NMR spectrometer using TMS as an internal reference. Elemental analyses were performed by Cairo-Elmer model 1240 CHN analyser.

Compounds 1a and 2a were obtained by reacting benzaldehyde with acetophenone and acetone respectively and 3a by reacting furfural with acetone by following standard procedures. Coumarin 4a was prepared from salicylaldehyde, sodium acetate and acetic anhydride.

Preparation of 5a - 8a. Acetophenone / acetone (3 mmole) was taken in a 50 mL round bottomed flask and 30 mL of methanol was added and stirred. To this 2 mL of 10% sodium hydroxide was added dropwise for a period of 10 min and the solution was kept stirring for 20 min at room temperature. To this 3-formyl-2-quinolone (3 mmole) was added and the reaction mixture was stirred for 4 hr. The mixture was poured into ice water. The solid obtained was filtered, washed with water, dried and recrystallised from methanol.

5a: Yield 50%, m.p. 224°C; IR(KBr): 3300 (-NH), 1660 (-CO-CH=CH-), 1640 (-CONH-); $^1$H NMR (CDCl$_3$): 8.5-8.31 (m, 12H, ethylenic, phenyl and aromatic protons), 10.21 (s, 1H, NH); Anal. Found: C, 78.39; H, 4.84; N, 5.09%.

6a: Yield 80%, m.p. 221°C; IR(KBr): 3400 (NH), 1680 (-CO-CH=CH-), 1648 (CONH) cm$^{-1}$; $^1$H NMR (CDCl$_3$): 8.05, 7.51, 4.79. Calcd for C$_{19}$H$_{13}$NO$_2$: C, 78.53; H, 4.76; N, 5.09%.

7a: Yield 66%, m.p. 226°C; IR(KBr): 3315 (NH), 1662 (-CO-CH=CH-), 1645 (CONH) cm$^{-1}$; $^1$H NMR (CDCl$_3$): 7.87, 5.23, 4.84%. Calcd for C$_{19}$H$_{13}$NO$_2$: C, 78.87; H, 5.23; N, 4.84%.

8a: Yield 74%, m.p. 264°C; IR(KBr): 3320 (NH), 1660 (-CO-CH=CH-), 1645 (CONH) cm$^{-1}$; $^1$H NMR (CDCl$_3$): 7.38, 5.58, 6.21. Calcd for C$_{19}$H$_{13}$NO$_2$: C, 73.99; H, 5.77; N, 6.16%.

General procedure for the reduction 1a - 8a using NaHTe. To a solution of NaHTe prepared in situ from tellurium powder 1.30 g (0.01 mole) and sodium borohydride 0.9 g (0.03 mole) in ethanol (50 mL) was added the substrate a (0.01 mole). The mixture was
Table I—Reduction using sodium hydrogen telluride

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<th>Substrate</th>
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IR spectra (KBr) of c. 5b - 8b showed ν<sub>max</sub> at 1695 - 1720 cm<sup>-1</sup> (–CO–CH<sub>2</sub>–CH<sub>2</sub>–); <sup>1</sup>H NMR spectra (CDCl<sub>3</sub>): 1a δ 2.9 - 3.4 (4H, coupled triplets), 7.4 - 7.7 (5H, m, ArH), 7.9 - 8.2 (5H, m, ArH); 5a δ 3.31 (t, 2H, CH–H), 3.94 (t, 2H, C2–H), 7.22 - 8.55 (m, 10H aromatic, phenyl protons), 10.51 (s, 1H, NH)

Satisfactory analytical d. data were obtained for all compounds.

refluxed for 4-5 hr under nitrogen, cooled, filtered, poured into ice cooled water/crushed ice. For 1a - 4a, product was extracted with chloroform. The organic layer was dried over anhyd. Na<sub>2</sub>SO<sub>4</sub>, evaporated and the residue was either purified by recrystallisation from ethanol or by distillation under reduced pressure. For 5a - 8a, the product obtained as solid was filtered and recrystallised from ethanol.

References