

The Synthesis of Dialdehyde from Sodium Hydrogen Telluride with 1,1',3,3'-Tetramethyl-2,2'-bis-benzimidazolium Salt

Ke Fen YUE*, Huan GU, Zhen SHI

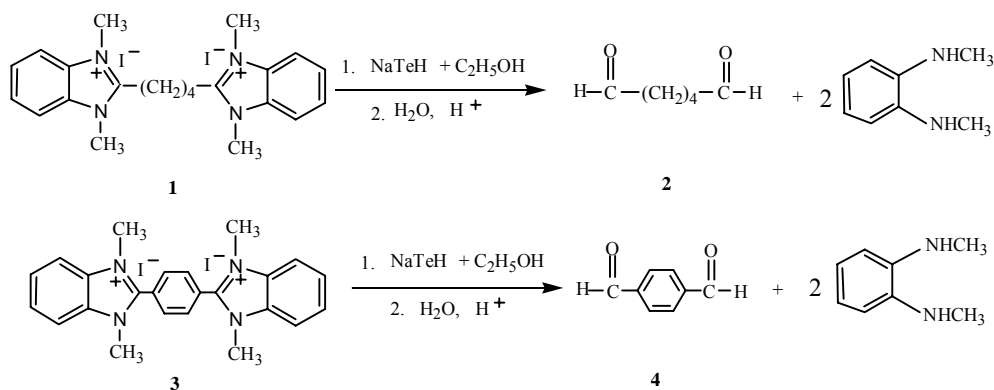
Department of Chemistry, Northwest University, Xi'an 710069

Abstract: The reaction of bis-benzimidazolium salts with sodium hydrogen telluride is reported, and a new synthetic method for dialdehydes is provided.

Keywords: Bis-benzimidazolium salt, sodium hydrogen telluride, dialdehyde, synthesis.

Dialdehydes are important chemical industrial materials. An efficient preparation method of dialdehydes has been interested. We have reported two novel synthetic methods of dialdehyde *via* the addition-hydrolysis reaction of benzimidazolium salt with bis-Grignard reagent¹ and the reduction-hydrolysis reaction of diimidazoline².

Sodium hydrogen telluride has been shown to be a convenient reagent for the reduction of unsaturated nitro-compounds to saturated ones³. The reduction of N-methylpyridinium iodide and N-methylisoquinolinium iodide by sodium hydrogen telluride was reported⁴. In this paper, we reduced bis-benzimidazolium salts by this reagent, and this provided a new approach to the preparation of dialdehydes. The route for the synthesis of dialdehydes is as follows:



*E-mail: ykflyy@263.net

Experimental

Sodium hydrogen telluride was prepared according to the literature⁵. 2,2'-Bis-benzimidazole was prepared from dicarboxylic acid with *o*-phenylenediamine in nearly quantitative yield⁶.

Synthesis of 1,1',3,3'-tetramethyl-2,2'-bis-benzimidazolium salts

1, 1', 3, 3'-Tetramethyl-2, 2'-bis-benzimidazolium salts were prepared in high yield as reported in literature⁷⁻⁸. Compound **1** and compound **3** were prepared respectively by similar method.

0.46 g of sodium cut in small pieces was added in 15 mL of anhydrous ethanol. After all the sodium was dissolved, 0.01 mol of 2,2'-bis-benzimidazole, 0.08 mol of iodomethane and 15.0 mL of benzene were added to this solution. The stirring was started and the mixture was refluxed for 18 h. The solvent was removed. The residue was recrystallized from ethanol to give pure compound **1**, lightly yellow powder, yield 85%, mp>300°C (lit.⁸ mp>300°C), while compound **3** was obtained by recrystallization from acetone. Compound **3** as brown powder, yield 80%, mp>300°C.

Synthesis of dialdehyde

1,1',3,3'-Tetramethyl-2,2'-bis-benzimidazolium salt (0.005 mol) was added in small portions to a solution of sodium hydrogen telluride (0.015 mol) in ethanol under nitrogen. After the mixture was stirred for 1.0-1.5 h at room temperature, 20 mL of saturated aqueous solution of oxalic acid was then added, and the mixture was heated at 40-50°C for 1.0 h with stirring. Ethanol was removed by distillation and the residue was extracted with ether or benzene (4×30 mL). The extracts were washed with 5% sodium bicarbonate, dried over anhydrous magnesium sulfate. After the solvent was evaporated, the residue was distilled to give compound **2**, while compound **4** was purified by recrystallization from water.

Compound **2** as colourless liquid, yield 66.8%, b.p. 92-94°C /1.999 kPa, n_D^{20} 1.4338 (lit.⁹ b.p. 92-94°C /1.999 kPa, n_D^{20} 1.4350); Compound **4** as white solid, yield 64.0%, mp 114-115°C (lit.¹⁰ 114-116°C).

Acknowledgments

This work was supported by the National Natural Science Foundation of China (No. 29872032).

References

1. Zhen Shi, Huan Gu, *Chin. Chem. Lett.*, **1997**, 8 (6), 465.
2. Zhen Shi, Huan Gu, Lili Xu, *Chin. Sci. Bull.*, **1997**, 42 (2), 130.
3. A. Osuka, *Bull. Soc. Chim. Japan*, **1985**, 58, 1067.
4. D. H. R. Barton, A. Fekin, X. Lusinchi, *Tetrahedron Lett.*, **1985**, 26 (31), 3693.

The Synthesis of Dialdehyde from 1,1',3,3'-Tetramethyl-2,2'-bis-benzimidazolium Salt

5. J. Chen, X. Y. Zhou, *Synthesis*, **1987**, (6), 586.
6. C. V. Prakash, *Chemistry & Industry*, **1980**, (5), 287.
7. V. A. V. El'tsov, K. L. Muravich, L. M. Roitshtein, *Zh. Org. Khim.*, **1967**, 3, 205.
8. S. N. Tremsin, *Farmakol Alkaloidov Ikh Proizvodnykh*, **1972**, 162.
9. R. C. Weast, *Handbook of Chemistry and Physics*, 63rd. Boca Raton, Florida: CRC Press Inc., **1982**, c-81.
10. Beijing Chemical Reagents Company, *The Handbook of Chemical Reagents Catalogue*. Beijing: Beijing Industry University Press, **1993**, 846.

Received 22 November, 2001