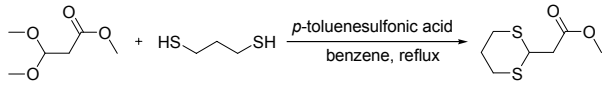
# Preparation of Bio-Functionalized Surfaces with an Electrochemically Active Silane Presenting Concealed Aldehyde†

**Hyejin Park, Al-Monsur Jiaul Haque, and Kyuwon Kim***

*Department of Chemistry, University of Incheon, Incheon 406-772, Korea. *E-mail: kyuwon_kim@incheon.ac.kr*
*Received September 14, 2010, Accepted October 6, 2010*

**Synthesis of 2-(1,3-dithiane-2-yl)-*N*-(3-(triethoxysilyl)propyl)acetamide.**

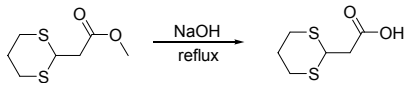
**Synthesis of A methyl 2-(1,3-dithiane-2-yl)acetate:**




To a solution of benzene (60 mL), 3,3-dimethoxypropanoate (5g, 0.0337 mM), propane-1,3-dithiol (3.39 mL, 1 eq), and *p*-toluenesulfonic acid (60 mg) was added, and then the mixture was heated under reflux for 25 h. Then the mixture was concentrated and the residue was distilled under lower pressure. Following distillation at 108 ~ 109 °C (0.3 Torr) gave the desired product in 30% yield.

$^1$H-NMR: δ 4.399-4.424 (t, 1H, CH), 3.721-3.731 (s, 3H, $CH_3$), 2.861-2.929 (m, 2H, $CH_2$), 2.787-2.799 (m, 4H, $CH_2$), 18899-2.117 (m, 2H, $CH_2$).

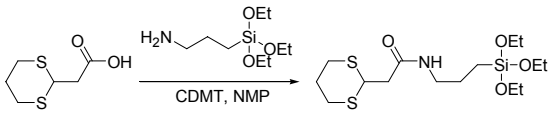
**Synthesis of 2-(1,3-dithian-2-yl)acetic acid:**




A mixture of 2-(1,3-dithian-2-yl)acetate (1.45 g, 7.54 mM), sodium hydroxide (0.6 g, 2 eq) and 10 mL of water was heated under reflux for 0.5 h. Then the mixture was cooled down to room temperature and acidified with concentrated HCl. The resulting mixture was filtered, followed by washing with chilled water. The mixture was dried in vacuum to give a white solid product (1.3 g, quantitative).

$^1$H-NMR: δ 4.26-4.4 (t, 1H, CH), 2.845-2.909 (m, 6H, $CH_2$), 1.9-2.12 (m, 2H, $CH_2$).

**Synthesis of 2-(1,3-dithian-2-yl)-*N*-(3-(triethoxysilyl)propyl)acetamide:**




A mixture of 2-(1,3-dithian-2-yl)acetic acid(0.9 g, 5.05 mM), CDMT (2-chloro-4,6-dimethoxy-1,3,5-triazine, 1.06 g, 1.2 eq), NMM (4-methylmorpholine, 1.53 g, 3 eq), and 15 mL of dry THF was stirred at room temperature for 1 h. The resulting mixture was added to 3-aminopropyl triethoxy silane (1.12 g, 1 eq), followed by heating under reflux overnight. After that, the mixture was cooled down to room temperature. Then aqueous solution of $NaHCO_3$ was added to the resulting reaction mixture. The reaction mixture was extracted with ethyl acetate, and the organic layers were dried over $Na2SO_4$ and filtered. Evaporation of the solvent gave light brown product. Further purification was achieved by recrystallization from Hex/EA and gave a white solid product in 62% yield.

$^1$H-NMR: δ 6 (s, 1H, NH), 4.484-4.508 (t, 1H, CH), 3.792-3.839 (m, 2H, $CH_2$), 3.271-3.293 (t, 2H, $CH_2$), 2.851-2.967 (m, 4H, $CH_2$), 2.543-2.555 (d, 2H, $CH_2$), 1.884-2.137 (m, 2H, $CH_2$), 1.622-1.672 (m, 2H, $CH_2$),1.202-1.247 (m, 9H, $CH_3$), 0.546-0.579 (t, 2H, $CH_2$).

†This paper is dedicated to Professor Hasuck Kim for his outstanding contribution to electrochemistry and analytical chemistry.